

DEVELOPMENT OF ADVANCED MATERIALS COMPOSITES FOR USE AS INSULATIONS FOR LH2 TANKS

CP-123928

SUMMARY REPORT

1 JULY 1971 TO 29 APRIL 1972

C. R. LEMONS
C. R. WATTS
O. K. SALMASKY

MDC G3677
JUNE 1972

(NASA-CP-123928) DEVELOPMENT OF ADVANCED
MATERIALS COMPOSITES FOR USE AS INSULATIONS
FOR LH2 TANKS Summary C.R. Lemons, et al
(McDonnell-Douglas Astronautics Co.) Jun.
1972 176 p

N73-11547

CSCI 11D G3/14 Unclass
16483

Prepared under Contract NAS 8-25973
by Materials and Methods - Research
and Engineering Department
McDonnell Douglas Astronautics Company
Huntington Beach, California
for the
National Aeronautics and Space Administration



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PREFACE

This documents reports the results of Phase II of the NASA Marshall Space Flight Center program entitled, "Development of Advanced Materials Composites for Use as Insulation for LH₂ Tanks." This work was conducted under NASA Contract NAS8-25973. Dr. J. M. Stuckey served as principal contracting officer representative. This summary report covers the period from 1 July 1971 to 29 April 1972.

The Program Manager was O. K. Salmassy. The Principal Investigator on this program was C. R. Lemons; he was supported by C. R. Watts. This program was under the overall technical direction of R. F. Zemer, Deputy Director, Materials and Methods - Research and Engineering.

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ABSTRACT

A study of internal insulation materials and fabrication processes for NASA Space Shuttle LH₂ tanks is reported. Emphasis was placed on an insulation system capable of reentry and multiple reuse in the Shuttle environment. Results are given on the optimization and manufacturing process scale-up of a 3D fiber-reinforced foam insulation, EX-251-3D, derived from the NASA/McDonnell Douglas Saturn S-IVB internal insulation.

It is shown that BX-251-3D can be satisfactorily installed in large-scale tanks under conditions that will permit a significant cost saving over the existing S-IVB technology.

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CONTENTS

Section 1	INTRODUCTION	1
	1.1 Objectives	1
Section 2	EXPERIMENTAL PROGRAM	3
	2.1 Literature Survey (Task 1)	5
	2.2 Foam Optimization (Task 2)	5
	2.2.1 Foam Mixing Machine Operation Procedure and Effect of Variations	12
	2.2.2 BX-251A-3D Weight Loss at Elevated Temperature	13
	2.2.3 BX-251A-3D Foam Density	17
	2.2.4 Tensile Bond Strength Using BX-251A-3D Foam	18
	2.2.5 Compressive Strength of BX-251A-3D Foam	20
	2.2.6 Shear Strength (Plate) of BX-251A-3D Foam	20
	2.2.7 Porosity of BX-251A-3D Foam	20
	2.3 Bonding Optimization (Task 3)	23
	2.3.1 Adhesive Mixing Verification	24
	2.3.2 Minimum Weight Adhesive for Bonding Foam to Tank Wall	32
	2.3.3 Catalyzed Working Life	37
	2.3.4 Cold Hold Time Under Vacuum Bag Pressure (Delayed Start) to Warm Cure	49
	2.3.5 Warm Cure Temperature Limits	51
	2.3.6 Vacuum Pressure Limits	57
	2.3.7 Postcure Limits	65
	2.3.8 Other Related Work	65
	2.4 Vibration and Acoustic Analysis	69
	2.4.1 Environmental Definition	72
	2.4.2 Fatigue Test Definition	82
	2.5 Panel Joints (Task 5)	90
	2.5.1 Thermal Analyses	90
	2.5.2 Structural Evaluation of Panel Joint Configurations	99
	2.5.3 Influence on Liner Strength by Filled Joints in 3D Foam	104
	2.6 Insulation Repair (Task 6)	108

	2. 6. 1	Methods for Applying Heat to Cure Repair Areas of Insulation	116
	2. 6. 2	Non-Destructive Examination of Bond Lines 3D Foam-to-Tank Wall, 211A/LZ Adhesive	118
	2. 6. 3	Examination of Debonds Between Glass Liner and 3D Foam, Epon 828/CL	120
	2. 7	Surface Preparation (Task 7)	124
	2. 8	Tanking Test Plan (Task 8)	127
Section 3		SPECIFICATION REQUIREMENTS	137
	3. 1	Foam	137
	3. 2	Bonding	137
Section 4		SUMMARY AND CONCLUSIONS	139
Section 5		REFERENCES	141
Appendix		SURFACE PREPARATION	143

FIGURES

1	Overall Program Approach	4
2	Fixture for Vacuum Assist Foam Fabrication	6
3	Comparison of 3D Foam Appearance	8
4	Collapsed 3D Foam	10
5	3D Foam Fabrication Setup	11
6	Weight Loss of BX-249N-3D and BX 251A-3D Foam	15
7	Stress-Strain Curves-Tension and Compression (Temperature 20°C) (68°F)	19
8	BX-251A Polyurethane Foam Shear Stress-Strain Curves (Z Direction)	21
9	Porosity Test Fixture	22
10	Elevated Temperature Test Fixture Tensile Bond Strength-Hot Plate	25
11	LH ₂ Tank Cure Cycle	26
12	Tank Wall Adhesive Mixing Machine	31
13	Tank Wall Adhesive Extruder	31
14	Lefkoweld 211A/LZ, 0.48 kg/m ² (45 grams/ft ²)	33
15	Plexiglas Plate Adhesive Flow Test	34
16	Plexiglas Plate Adhesive Flow Test	34
17	Zeiss Opton Abbe Refractometer	38
18	Index of Refraction — Temperature Correction Graph for EPON 828/CL Resin	39

19	Catalyzed Working Life of Lefkowied 211A/LZ Adhesive	44
20	Liner Resin Flow Test	47
21	Catalyzed Working Life of EPON 828/CL Liner	50
22	Intermittent Loss of Vacuum Pressure During Warm Cure Cycle	62
23	Tank-Wall Adhesive Lefkowied 211A/LZ Postcure	67
24	EPON 828/CL Liner Postcure	68
25	Liner Burst Test Setup	70
26	Liner Burst Test Panel Configuration	71
27	Configuration of Shuttle Booster Liquid Hydrogen Tank	74
28	Resonant Frequencies of LH ₂ Tank During Liftoff	76
29	Resonant Frequencies of Upper Half of LH ₂ Tank During Maximum Dynamic Pressure ²	77
30	Resonant Frequencies at Reentry	78
31	Predicted Acoustic Sound Pressure Levels for Shuttle Booster Hydrogen Tank	79
32	Dynamic Amplification Factor Versus Resonant Frequency of Booster LH ₂ Tank Used in Analysis	80
33	Acceleration Response at the Center and Edge of Shuttle Booster Hydrogen Tank at Liftoff	80
34	Maximum Acceleration PSD's on Tank Wall During Maximum Dynamic Pressure	81
35	Acceleration Response at Center and Edge of Tank for Reentry Condition	81
36	Maximum Stress PSD's in Tank Wall During Liftoff	83

37	Maximum Stress PSD's in Tank Wall During Maximum Dynamic Pressure	93
38	Schematic of Beam Coupon Specimens	86
39	Fatigue Curve	88
40	Mission Life Blocks	89
41	Panel Configuration for Acoustic Tests	90
42	Joint Configurations	92
43	3-D Reinforced Saturn Foam—Maximum Thermal Conductivity	93
44	Shiplap Joint	96
45	Butt Joint	97
46	Radiation Heat Transfer Contribution Shiplap Joint	98
47	Horizontal Temperature Distribution Shiplap Joint	99
48	Beam Column Analysis Model	100
49	Typical Deflected Shape of Liner	101
50	Bond Line Tensile Stress at Edge of Unsupported Liner	102
51	Finite Element Model	104
52	Joint Test Configuration 3D Foam	106
53	Test Setup for Joint	107
54	Joint Contraction Specimens After LN ₂ Test	108
55	LN ₂ Contraction Tests BX-251A-3D Bonded Joint	109
56	Typical Repair Procedure—Small Damaged Area	111
57	Sponge Pressure—Small Repair Procedure	112
58	Vacuum Bag Pressure During Cure (Repair)	116
59	Ultrasonic Examination of 3D Foam-to- Tank Wall Bond	119

60	Ultrasonic Patterns from Reference Panels 211A/L2 Adhesive to Aluminum	121
61	Sonic Brush	122
62	Test Tank—2.4 m (8 Ft)	130
63	Temperature Sensor Location—2.4 m (8 Ft) Scale Tank	132
64	Subscale Tank Test	133

TABLES

1	Properties of Foam Blocks	14
2	Tensile Strength After 177 °C (350 °F) Exposure	17
3	Evaluation of Methods for Determining Adhesive Mix Thoroughness	28
4	Degree of Adhesive Mixing	29
5	Mixture Ratio Variation	30
6	Machine-Mixed Adhesive	33
7	Bond Strength Using Low-Weight 211A/LZ Adhesive Application	36
8	Liner Contraction Tests	40
9	Tank Wall Adhesive Hold Under Vacuum Bag Pressure	52
10	Liner Hold Under Vacuum Bag Pressure	53
11	Liner Strength After Cure Without Vacuum Bag	54
12	Low-Pressure Limit of Tank Wall Adhesive Low Limit of Vacuum Bag Pressure During Bonding of 3-D Foam to Tank Wall	58
13	Low-Pressure Limit of Liner	59
14	Specimen Cure History Intermittent Loss of Vacuum Pressure Test	61
15	Tank Wall Adhesive Intermittent Loss of Vacuum Pressure After 25 °C (77 °F) Hold	63
16	Tank Wall Adhesive Intermittent Loss of Vacuum Pressure After 2 °C (35 °F) Hold	63
17	Liner Intermittent Loss of Vacuum Pressure After 25 °C (77 °F) Hold	64

18	Liner Intermittent Loss of Vacuum Pressure After 2°C (35°F) Hold	44
19	Time Between Warm Cure and Postcure of Tank Wall Adhesive	66
20	Time Between Warm Cure and Postcure of Liner	66
21	Environmental Definition Factors	73
22	Equivalent Limit and Fatigue Dynamic Stresses in the Shuttle Booster Hydrogen Tank Cylinder	84
23	Initial Fatigue Stress Levels in the Glass Cloth Liner for the Beam Bending Coupon Tests	87
24	Predicted Initial Values of Oscillating Force to be Applied to First Beam Specimens	87
25	Acoustic Levels for Panel Tests	91
26	3-D Foam Thermophysical Properties	95
27	Effect of Sponge Pressure During Cure on Double Layer 211A/LZ (Repair)	114
28	Effect of Sponge Pressure During Cure on Single Layer 211A/LZ (Repair)	115
29	Effect of Sponge Pressure During Cure on Epon 828/CL Glass Liner (Repair)	115
30	Anodize Study	125

Section 1 INTRODUCTION

1.1 OBJECTIVES

The primary objective of this program has been to develop reliable advanced material composites for a minimum-weight internal insulation that will provide adequate protection of NASA Space Shuttle LH_2 tanks for up to 100 and preferably 200 missions. Emphasis has been placed upon developing a concept that will not be adversely affected by LH_2 tank outer-surface temperatures of up to 177°C (350°F).

The basic approach of this program has been to develop a modification of the MDAC/NASA-developed S-IVB-3D foam insulation for application to the more severe environment of the Space Shuttle. Phase I of this program was successful in developing the basic material. An improved composite was developed, and its performance was verified.

The baseline composite selected from Phase I of this program consisted of three-dimensionally (3-D) reinforced, heat-stabilized BX-249N foam, with a liner of 828/CL resin-impregnated, 116-glass cloth and a tank-wall adhesive of L211A/LZ resin.

Objectives of Phase II included the optimization of the baseline composite and analysis of Space Shuttle vehicle environments not considered previously. Tank insulation fabrication and installation procedures which are directly applicable to the Space Shuttle were established. During Phase II, an as-machined metal surface with a chromic-acid anodized coating was selected as the baseline tank-wall condition (Task 7). During Phase II as-molded BX-251A-3D foam with silane (EC 3901) primed fibers was substituted for BX-249N-3D-S as the baseline reinforced foam system.

Because of the functional similarities between S-IVB and Shuttle internal insulation requirements, the performance requirements established for the S-IVB system were used during Phase I as the basis for material developments.¹ Phase B Shuttle studies were subsequently completed along with extension studies.²

During the Shuttle Phase B studies, emphasis was shifted from a completely reusable system to analysis of an Orbiter with external expendable tanks;³ under a recent expendable tank concept, the Orbiter carries its hydrogen propellant in one external tank. The tank would have a smooth wall with isogrid reinforcement in some areas. None of the insulation requirements set forth in Reference 1 was altered for this study by these more recent system considerations.

Section 2

EXPERIMENTAL PROGRAM

The adhesive, 3D foam, and liner materials that were developed under Phase I of this contract have demonstrated, through subscale (dome) simulation testing, the basic capability to meet the Space Shuttle operational requirements. Since the resulting insulation system is a direct derivative of the successful S-IVB-3D foam system in concept, makeup, and operational principle, significant confidence exists in the ability to achieve successful manufacturing scaleup and large-tank performance. Phase II of this program was, therefore, devoted to development and demonstration of insulation fabrication and installation procedures suitable for large-scale Shuttle tank application. In addition, studies were conducted to evaluate the adequacy of insulation construction, joint design, and repair approaches. At the conclusion of Phase II, MDAC prepared a simplified test plan for subscale tank verification testing—the last step, as established by the Saturn S-IVB program, in the qualification of an internal insulation for large-tank application. The overall program approach is described in Figure 1.

Initial effort was directed at Task 7, Surface Preparation; Task 3, Bonding Optimization; and Task 2, Foam Optimization. In the latter task, some effort was carried over from Phase I, Task 4, Methods of Decreasing Weight, as an approach to foam optimization. Tasks 4 and 5 were initiated last to allow use of as well-developed Shuttle environment inputs as possible.

The program was divided into the following tasks:

- Task 1—Literature Survey
- Task 2—Foam Optimization
- Task 3—Bonding Optimization
- Task 4—Vibration and Acoustics Analysis
- Task 5—Panel Joints
- Task 6—Insulation Repair

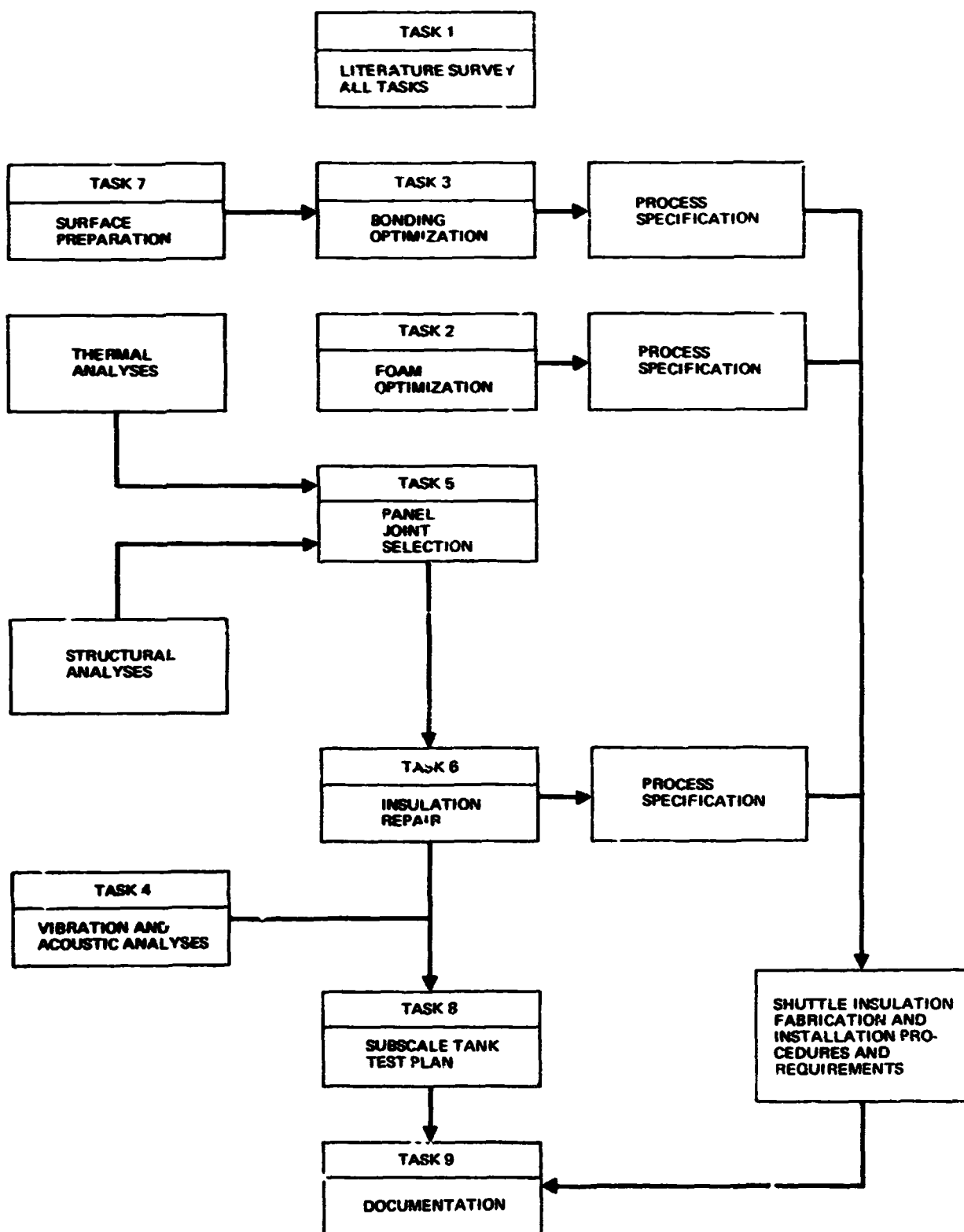


Figure 1. Overall Program Approach

Task 7 – Surface Preparation

Task 8 – Tanking Test Plan

2.1 LITERATURE SURVEY (TASK 1)

Objective of this task was to conduct a comprehensive, up-to-date survey of the technical literature dealing with cryogenic insulation, low-heat-conductive materials, and other related topics. Pertinent information sources have been reported in previous quarterly and monthly contract documentation.

2.2 FOAM OPTIMIZATION (TASK 2)

Objectives of this task were to determine optimum 3-D foam fabrication and processing conditions and to determine the variations from optimum conditions that were allowable.

As a result of the Phase I effort, heat stabilized 3D reinforced NOPCO BX-249N foam was selected as the baseline foam for Phase II. BX-249N foam exhibits a free rise density of about 0.032 gm/cm^3 (2 lb/ft^3). When fabricated using the standard S-IVB-3D thread reinforcement and a mold preheated to about 93°C (200°F), the 3D reinforced foam composite has a density of about 0.056 gm/cm^3 (3.5 lb/ft^3). After heat stabilization for 16 hours at 177°C (350°F) the bulk density is about 0.048 gm/cm^3 (3 lb/ft^3).

The unstabilized 3D reinforced foam is designated BX-249N-3D. The heat stabilized 3D reinforced foam is designated BX-249N-3D-S.

In order to optimize the insulation system weights, techniques were explored for reducing the 3D foam density. A series of 3D blocks were fabricated by a process in which the foam was allowed to rise through the 3D fiber array while the assembly was under vacuum pressure instead of using the normal fabrication operation conducted at ambient atmospheric conditions. The test fixture used for fabrication of vacuum assist rise 3D foam blocks is shown in Figure 2, and the procedure used is described below:

- A. The 41 cm (16-inch) cube vacuum chamber shown in Figure 2 also served as a mold to contain the foam during the rise period. This chamber, and fiber array supported inside aluminum frames, was sealed and leak checked prior to introducing the foam.

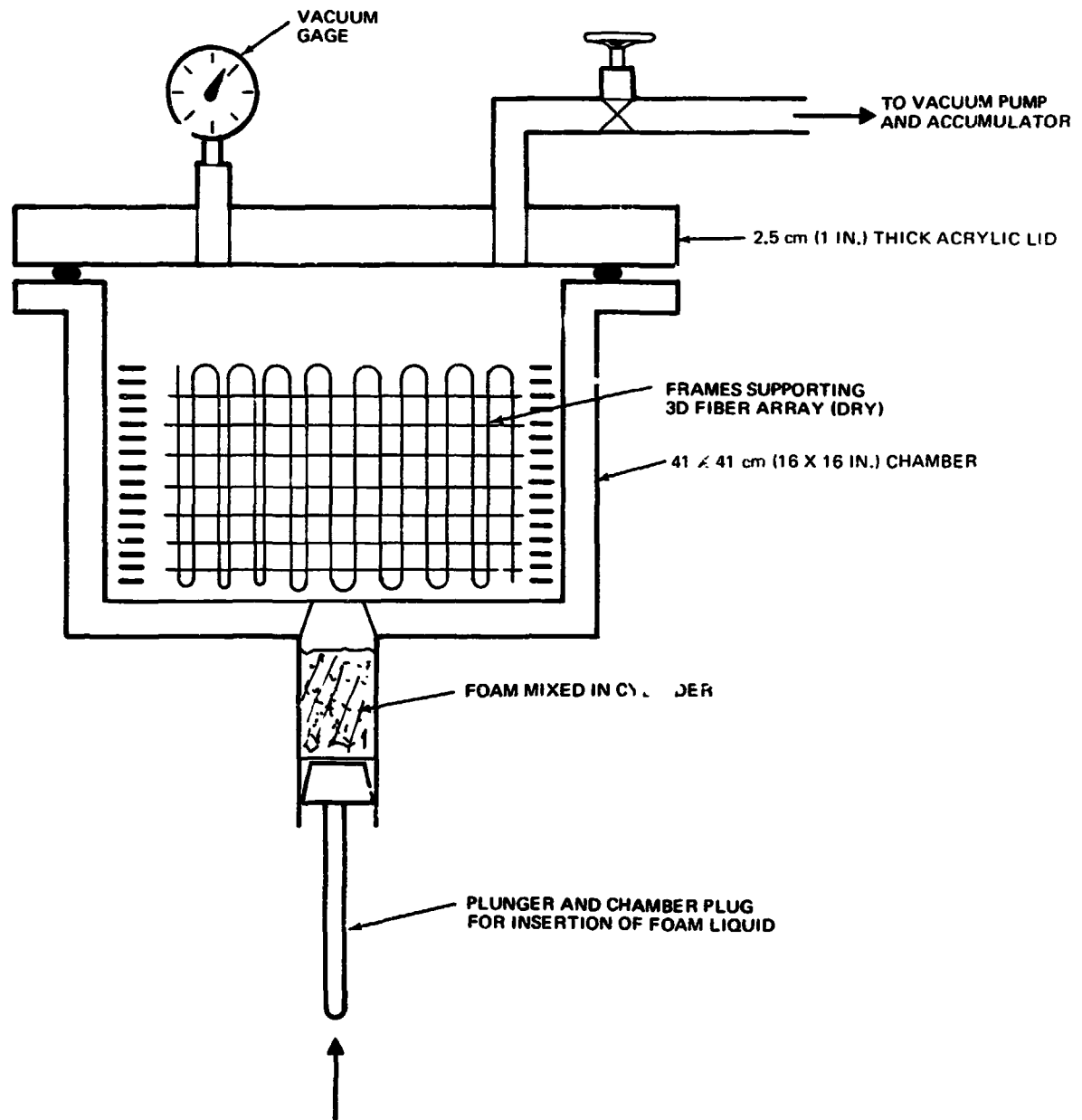


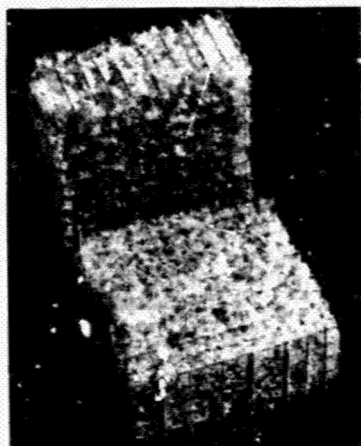
Figure 2. Fixture for Vacuum Assist Foam Fabrication

- B. The chamber and fiber array were preheated in an oven at 150°C (300°F) for 2 hours then removed from the oven.
- C. An acrylic lid was sealed over the chamber top opening.
- D. The foam, BX-249N, was mixed manually using a motor-driven stirring blade for a time period of 8 seconds, then immediately injected into the chamber through an orifice in the bottom of the air chamber. The orifice, 3.8 cm (1.5 in.) in diameter, was instantly plugged with the foam injection plunger. The chamber temperature at foam injection was 94°C (200°F).
- E. The air within the chamber was evacuated rapidly using an accumulator vessel so as to achieve vacuum pressure of 0.084 MN/m² (25 inches of mercury) within 25 seconds (rise time with BX-249N foam is ~60 seconds).
- F. This vacuum pressure was maintained until the foam developed sufficient stiffness to prevent cell collapse when the pressure was returned to 1 atmosphere. This "cure" time was ~5 hours.

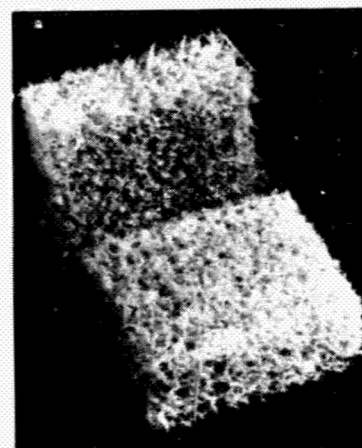
Preliminary tests showed that, to be effective in reducing the density of the foam, the vacuum pressure must be applied before the mixed foam begins to rise or at least within the first half of the foam rise time which is 60 seconds for BX-249N foam. Unreinforced foam will respond to vacuum pressure as low as 0.061 MN/m² (18 inches of mercury) producing densities as low as 6.9 kg/m³ (0.43 lb/ft³).

The 3D fiber array offered considerable resistance to foam rise and a vacuum of 0.084 MN/m² (25 inches of mercury) was necessary to achieve composite density of 30 kg/m³ (1.9 lb/ft³). The larger foam cell size and quality of vacuum-assisted 3D foam is compared to BX-249N-3D foam in Figure 3.

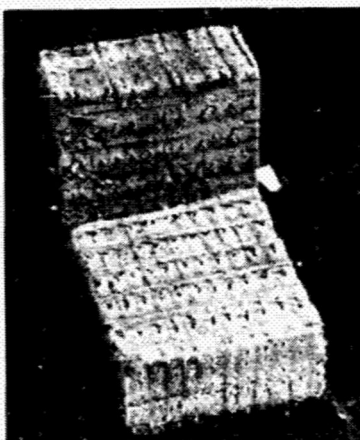
The very light impregnation of 3D fibers achieved with "vacuum assist" foam in these 3D foam blocks resulted in extremely low compressive strength (0.12 MN/m²; 17 psi). [A compressive strength of 1.2 MN/m² (170 psi) is achieved with Saturn S-IVB 3D foam at 83 kg/m³ (5.2 lb/ft³) density; however, since the compressive strength is a characterization property, not a design requirement on functional performance, a value lower than 1.2 MN/m² (170 psi) may be tolerated to achieve a lower weight composite.]



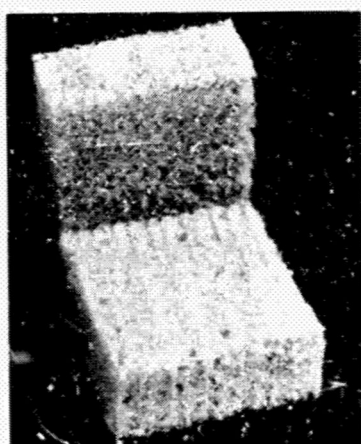
VACUUM ASSIST
3D FOAM BX-249N
PREPREG STIFF FIBERS
 35 kg/m^3 (2.2 PCF)
DENSITY



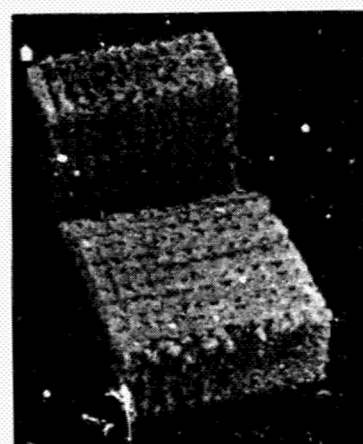
VACUUM ASSIST
3D FOAM BX-249N
NO FIBER PREPREG
 40 kg/m^3 (1.9 PCF)
DENSITY



3D BX-249N FOAM
HEAT STABILIZED
 48 kg/m^3 (3.0 PCF)
DENSITY



3D BX-251 FOAM
BEFORE HEAT STAB.
 40 kg/m^3 (2.5 PCF)
DENSITY



3D BX-251 FOAM
HEAT STABILIZED
 37 kg/m^3 (2.3 PCF)
DENSITY

Figure 3. Comparison of 3D Foam Appearance

The compressive strength of vacuum assist 3D foam blocks was subsequently increased by coating the fibers with epoxy resin, prior to introducing foam. A mixture of 1 part epoxy 828/CL resin to 2 parts acetone was sprayed into the dry 3D fiber array. After air drying the array for 2 hours the impregnation was given an oven cure of 1 hour at 135°C (275°F). At this stage the glass fibers were stiff, self-supporting, and possessed a resin content of 30 percent by weight. The foam was then introduced and the vacuum assist rise method previously described was used to produce a composite density of 35 kg/m³ (2.2 lb/ft³).

Weight of Dry 3D Fibers	11 (0.7)
Weight of Epoxy Resin Preimpregnation	5 (0.3)
Vacuum Assist Foam	<u>19 (1.2)</u>
Composite Density	35 kg/m ³ (2.2 lb/ft ³)

The 3D foam blocks made with preimpregnated fibers developed a compression strength of 0.59 MN/m² (85 psi). Slices of 3D foam 2.5 cm (1.0 in.) thick taken from these low density blocks were then prepared for bonding to aluminum plates and for bonding to the glass liner for the measurement of tensile bond strength. During the initial bonding operation at 52°C (125°F) in which the vacuum bag pressure used was 0.07 to 0.08 MN/m² (20 to 24 inches of mercury), these low density 3D foam cores all collapsed as shown in Figure 4. The foam was simply too weak at densities of 19 kg/m³ (1.2 lb/ft³) to provide support for the fibers. The sandwich construction moved laterally under the vacuum bag to collapse although many of the Z fibers within the 3D foam were not crushed.

The vacuum assist procedure was set aside and other techniques were explored to achieve low density along with structural acceptance.

A foam, Nopco BX-251A, had been reported by NASA as exhibiting superior thermal stability and excellent cell structures at densities lower than Nopco BX-249N. Evaluation and optimization of the NASA suggested BX-251A-3D was conducted independently by MDAC as part of its IRAD program on Shuttle HO tank external insulation.

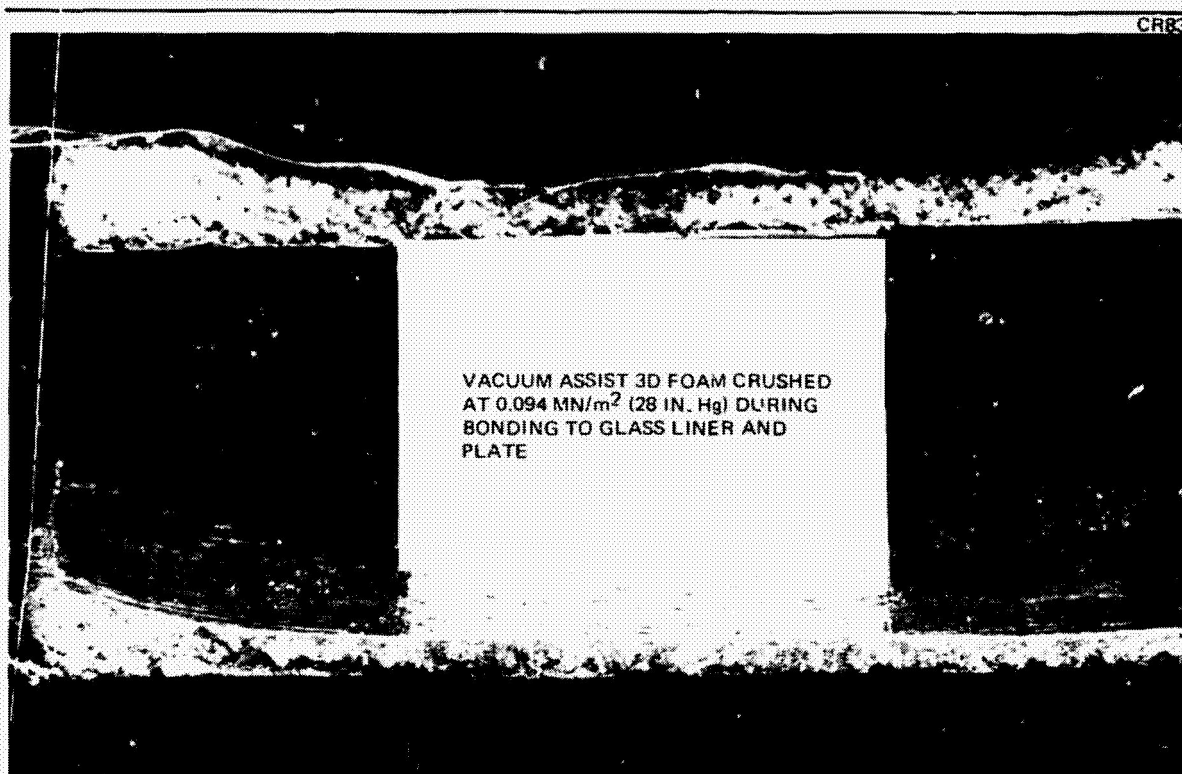


Figure 4. Collapsed 3D Foam

As a result of this independent optimization, MDAC, with the approval of the NASA COR, decided to substitute BX-251A-3D for the remainder of Phase II. The information provided below on foam process optimization is on BX-251-3D because of the pertinence to subsequent Phase II work.

Production type foam mixing and dispensing equipment shown in Figure 5 was used to pour a series of 33 blocks, 21 x 32 x 32 cm (8 x 12 x 12 inches) in size, of 3D foam using BX-251A foam. The first seven blocks, having untreated 3D fiber arrays, were used to establish the optimum mixing and pouring conditions outlined below. The results from varying the process conditions are also noted.

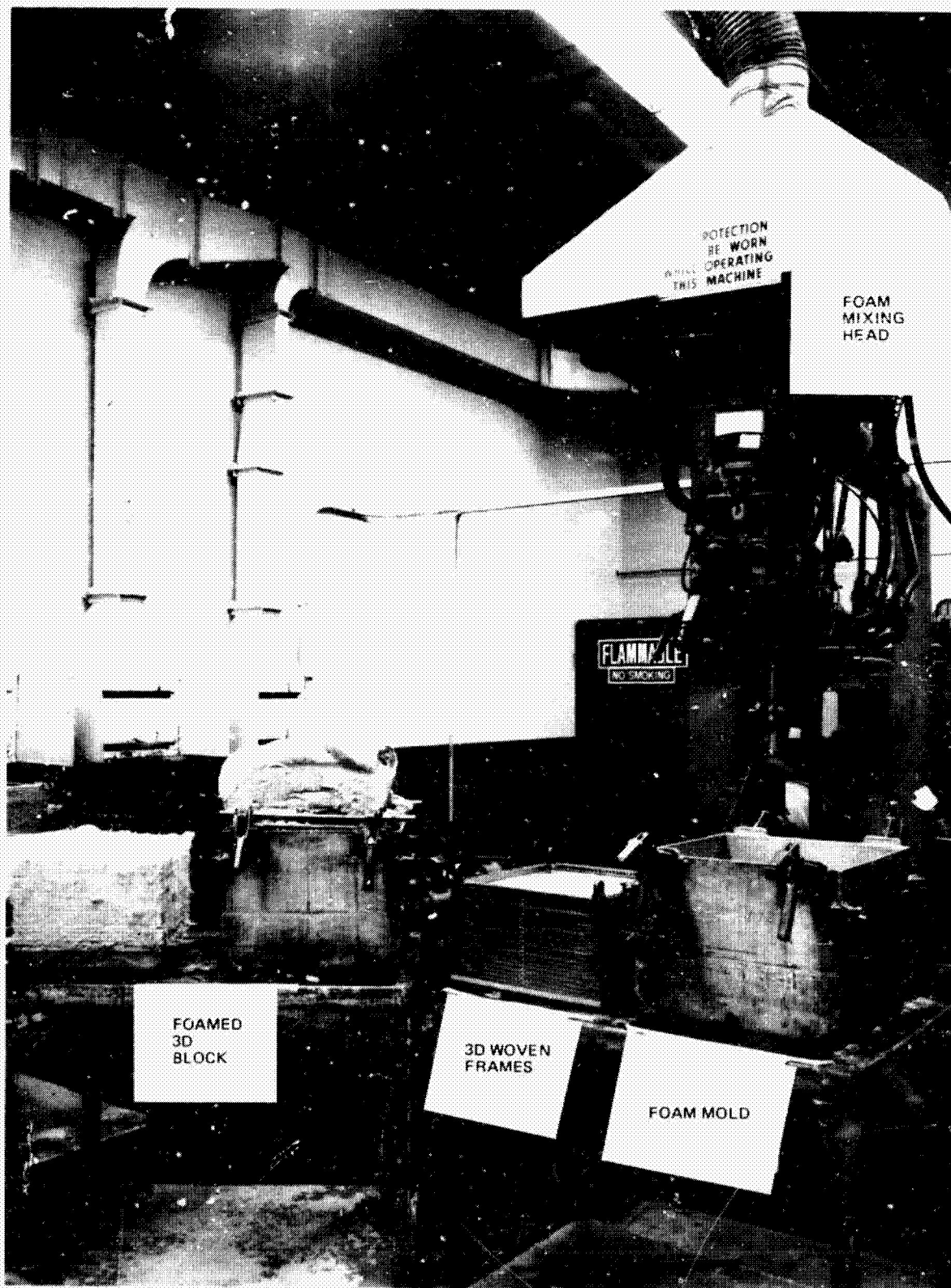


Figure 5. 3D Foam Fabrication Setup

2.2.1 Foam Mixing Machine Operation Procedure and Effect of Variations

- * 1. Stabilize foam ingredients to $28 \pm 2^{\circ}\text{C}$ ($80 \pm 3^{\circ}\text{F}$) and recirculate for 1 hour prior to calibration pouring.
 - Lower conditioning temperature, 21°C (70°F), resulted in slow foam rise and incomplete filling of the mold.
 - Higher conditioning temperature, 45°C (95°F), resulted in foam rise so rapid the 3D array cannot be positioned.
- * 2. Adjust hydraulic pump pressures to achieve a ratio of 41R to 59T within $\pm 2.5\%$ "T". Typical calibrations are 440 grams R to 633 grams T with a 5 second calibration pour (weigh each component separately).
 - The maximum variation measured during a 20 block run was $\pm 2.4\%$ or ± 5 grams in the 633 gram weight of the "T" component.
 - 3D blocks poured using a predetermined off-ratio mix of +10% and -10%, or ± 63 grams in the nominal 633 gram calibration weight, were judged to be of marginal quality relative to blow hole frequency and cell uniformity.
- 3. Adjust pour timer for a 10 second pour. This will deliver 2146 grams of liquid foam into the mold.
- * 4. Stabilize mold temperatures and fiber array at $27 \pm 2^{\circ}\text{C}$ ($80^{\circ} \pm 3^{\circ}\text{F}$).
 - Mold temperature of 66°C (150°F) resulted in the lowest density, 40 kg/m^3 (2.5 lb/ft^3), but very poor cell uniformity and numerous blow holes. The rise time was decreased to 35 seconds from a normal 65 seconds.
 - Mold temperatures of 18°C (65°F) resulted in a thick, 0.15 cm (0.06 in.), rubbery skin remaining on the mold surfaces and incomplete filling of the 3D array.
- * 5. Use blending rotor speed of 5,000 to 6,000 rpm.
 - Blending rotor speed of 3,000 rpm, or one-half the normal 6,000 rpm, was used during the pour of two blocks, one neat and one reinforced 3D. These blocks exhibited a slower rise time and visible streaks of unmixed ingredients in the liquid

*Indicates requirement items. Other notes are for information.

foam pool, but were of acceptable quality after cure. An rpm of 4,000 was required to eliminate streaks in the liquid.

6. Dispense 2146 grams of foam with a 10 second pour onto a polyethylene pan located just below the outlet nozzle so that air does not become entrained in the blended foam. Then lower the filled pan to rest against the bottom of the mold.
7. Place the fiber array on frames over the mold and clamp in position.
8. The foam will rise to the top of the fiber array (25.4 cm, or 10 inches, in height) within 65 seconds \pm 15 seconds and complete the rise period within 120 seconds \pm 20 seconds. The exothermic heat as measured in the center 5.1 cm (2 inches) from the bottom will reach $71 \pm 6^\circ\text{C}$ ($160^\circ \pm 10^\circ\text{F}$).
- * 9. Disassembly of the mold can occur 15 minutes after pouring.
 - Within 5 minutes after pouring the foam surface is tack free but is still soft. After 15 minutes the foam is cured to the extent that mold separation will be clean.
- * 10. Slicing of 3D foam can occur 8 hours after pouring.
 - Slicing the 3D foam block while the internal temperature, from exothermic reaction, is over 66°C (150°F) will result in contour instability. An 8 hour minimum cooling period is recommended.

Anticipating a very low compression strength condition with these 3D foam blocks using foam less than 32 kg/m^3 (2.0 lb/ft^3) density, the fiber arrays in other 3D blocks were preimpregnated and cured prior to foam injection.

Rigid epoxy resin solutions in acetone were used to preimpregnate the fiber array. Other blocks were made with the array coated with a silane primer, EC-3901, in an effort to improve the bond between glass fiber and foam. These blocks are listed in Table 1 along with their densities and summary of their strength properties. All BX-251 3D property data were obtained as a direct output of Task 2 efforts.

2.2.2 BX-251A-3D Weight Loss at Elevated Temperature

The weight loss at elevated temperature presented in Figure 6 shows the BX-251A-3D foam weight will decrease only 6% after 16 hours at 177°C

*Indicates requirement items. Other notes are for information.

Table 1
PROPERTIES OF FOAM BLOCKS

Type of 3D Foam	Low Temperature Test -196°C (-320°F)		High Temperature Test Holding 0.7 MN/m ² (100 psi)		Compression Strength 25°C (77°F) MN/m ² (psi)
	Metal Bond 211A/LZ MN/m ² (psi)	Liner Bond 828/CL MN/m ² (psi)	Metal Bond 211A/LZ °C (°F)	Liner Bond 828/CL °C (°F)	
BX-251A-3D 44 kg/m ³ (2.75 PCF) As molded	2.14 (311) 2.17 (315) 2.21 (320)	1.14 (165) 1.33 (200) 1.17 (170)	171 (340) 166 (330) 164 (328)	177 (350) 177 (350) 149 (300)	0.43 (62)
BX-251A-3D 40 kg/m ³ (2.5 PCF) Heat stabilized 16 hr - 177°C (350°F)	2.02 (293) 1.93 (280) 2.07 (300)	1.02 (148) 1.24 (180) 1.31 (190)	170 (338) 174 (344) 168 (334)	 143 (290)	0.50 (73)
BX-251A-3D EC-3901 Primed 3D 44 kg/m ³ (2.75 PCF) as molded	1.78 (258) 1.90 (275) 1.73 (250)	1.47 (213) 1.31 (190) 1.51 (219)	176 (348) 177 (350) 182 (360)	179 (355) 174 (346) 170 (338)	0.45 (65)
BX-251A-3D EC-3901 Heat stabilized 16 hr - 177°C (350°F)	1.74 (252) 1.59 (230) 1.42 (205)	1.49 (215) 1.64 (238) 1.68 (243)	164 (328) 177 (350) 	160 (320) 151 (304) 	0.43 (62)
BX-251A-3D 48 kg/m ³ (3.0 PCF) Epoxy impregnated 3D as molded	2.38 (345) 2.35 (341) 2.58 (374)	1.48 (215) 1.63 (236) 1.00 (145)	170 (338) 176 (348) 171 (340)	 151 (303)	0.68 (98)
BX-251A-3D 44 kg/m ³ (2.75 PCF) Epoxy impregnated 3D heat stabilized 16 hr - 177°C (350°F)	1.55 (225) 0.99 (143) 1.66 (240)	0.79 (115) 1.26 (183) 	178 (352) 178 (353) 	 150 (302)	0.61 (89)
BX-249N-3D 48 kg/m ³ (3.0 PCF) Heat stabilized 16 hr - 177°C (350°F)	2.07 (300) 1.99 (288) 1.90 (275)	1.64 (238) 1.59 (230) 1.82 (263)	169 (336) 174 (344) 172 (342)	164 (328) 153 (308)	0.35 (53) (0.60 MN/m ² ; 87 psi unstab)
Saturn S-IVB Min value CPR-30-3-3D	0.69 (100)	0.69 (100)	N/A	N/A	0.69 (100)
Min average 83 kg/m ³ (5.2 PCF)	1.04 (150)	1.04 (150)			
Typical average	1.72 (250)	1.38 (200)			1.22 (177)

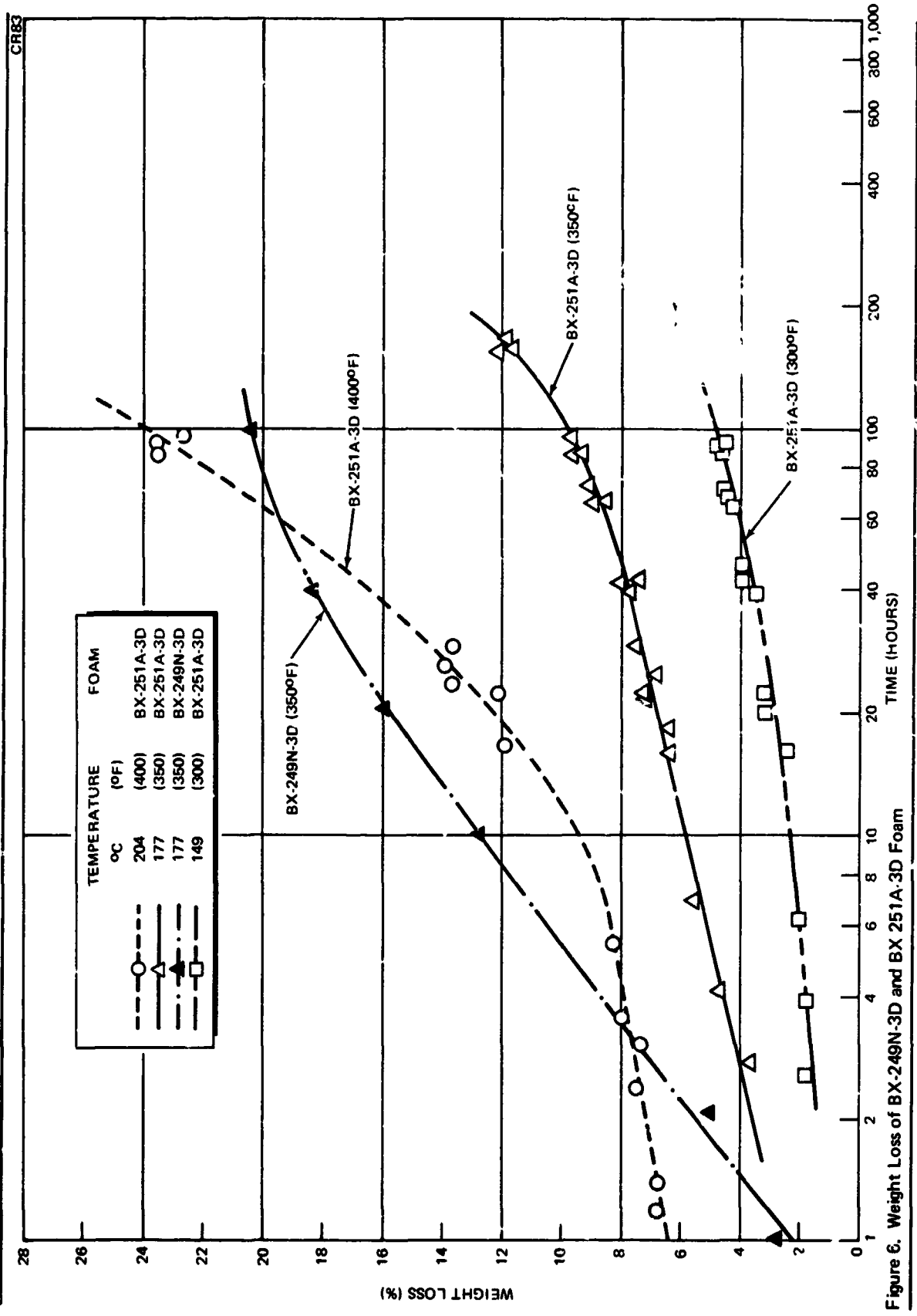


Figure 6. Weight Loss of BX-249N-3D and BX-251A-3D Foam

(350°F) exposure compared to 15% weight decrease for BX-249N-3D. Also the dimensional change, first expansion then shrinkage when exposed to 177°C (350°F), did not occur with BX-251A-3D. These factors indicated the heat stabilization cycle of 16 hours at 177°C (350°F) applied to BX-249N-3D throughout this program may not be required with the use of BX-251A-3D. The dimensional change, swelling, associated with BX-249N-3D has been sufficiently severe to rupture the liner bond when the sandwich was exposed to 149°C (300°F) during the post cure cycle of the adhesive. For this reason, BX-249N-3D panels have all received a heat stabilization treatment prior to bonding the sandwich with adhesives that require elevated temperature, no pressure, cure.

To confirm that BX-251A-3D would not require this heat stabilization cycle, test panels were fabricated using as-molded BX-251A-3D bonded to anodized 2219-T87 with Lefkowitz 211A/LZ. The same panels also received the 116 glass fabric liner impregnated and bonded with EPON 828/CL resin to complete the sandwich construction. Identical panels were also prepared using BX-249N-3D foam core that was not given the heat stabilization cycle prior to bonding the sandwich.

Both sets of panels were cured at 52°C (125°F) under vacuum bag pressure then postcured at 149°C (300°F) without the vacuum bag. No bond ruptures were visible at this point although the BX-249N-3D panels were swollen to produce a pillowed effect on the liner surface. The panels were then heated to 177°C (350°F) for 1 hour and reexamined. The pillowed condition on the liner bonded to the BX-249N-3D panel had receded but was still visible. No dimensional change was visible on the BX-251A-3D panels throughout this heating cycle. Both sets of panels were then cut into specimens for tensile bond strength determinations. The results of these strength tests are given in Table 2. The strength values associated with the pillowed liner bond to BX-249N-3D are low and show inconsistent bond strength but are higher than expected — previous panels of this type exposed to 177°C (350°F) swelled and ruptured the liner bond or the aluminum bond without reaching the test specimen preparation stage. The strength values associated with the BX-251A-3D panels tested at elevated temperature and at cryogenic temperature were as high as any values obtained with heat stabilized BS-249N-3D

Table 2
TENSILE STRENGTH AFTER 177° C (350° F) EXPOSURE^a

Foam Material	Average Tensile Strength					
	Tested at -196° C (-320° F)				Ruptured Under Stress of 0.7 MN/m ² (100 psi)	
	Tank Wall Adhesive	Liner		Tank Wall Adhesive	Liner	
	MN/m ² (psi)	MN/m ²	(psi)	° C (° F)	° C (° F)	° C (° F)
BX-249N	All ruptured on liner bond side of specimen	0.88	(127)	163	(325)	148 (298)
BX-251A		1.05	(152)	175 (347)	132 (360)	

^aUsed 3D foam which had not been heat stabilized and which had liner on one side and anodized 2219-T87 bonded to the other. Postcured for 16 hr at 149° C (300° F) then exposed 1 hr at 177° C (350° F) then another 16 hr at 149° C (300° F).

foam bonds, and showed no deleterious effects from not heat stabilizing the foam prior to bonding the sandwich.

Accordingly, with COR approval, contract-funded tests using BX-251A-3D foam were conducted on "as molded" slices and were not heat stabilized prior to adhesive bonding operations.

2.2.3 BX-251A-3D Foam Density

The density of BX-251A-3D foam blocks made without resin preimpregnation of the fiber array ranged from a low of 40.9 kg/m³ (2.56 lb/ft³) to a high of 46 kg/m³ (2.9 PCF) with a nominal of 43 kg/m³ (2.7 PCF). By subtracting the calculated weight of the dry glass fiber 3D array of 11 kg/m³ (0.7 PCF), the weight of the foam would be 32 kg/m³ (2.0 PCF). This same foam batch mixed by machine and poured into the mold without reinforcing fibers and allowed to rise without restriction developed a density of 17.8 kg/m³ (1.11 PCF) to 20.2 kg/m³ (1.26 PCF). Therefore, the densification of the foam created by the 3D fiber array restriction to the rise was only ~13 kg/m³ (~0.8 PCF). Further efforts to reduce the density by modifying the manufacturing process would be confined to ~17 kg/m³ (~1.05 PCF) and would

quickly reach a point of diminishing returns. For this reason, the composite density was established at $43 \pm 5 \text{ kg/m}^3$ ($2.7 \pm 0.3 \text{ PCF}$) for BX-251A-3D foam blocks.

The BX-251A foam, as received from the manufacturer, Diamond Shamrock Corp., would be tested by the quality assurance laboratory using a rotor blade mixing method prior to production acceptance for use in the mixing machine. For this reason a series of neat foam blocks were fabricated and tested to establish proposed specification values applied to this foam. The results of these tests on neat laboratory mixed, BX-251A foam are given in Figure 7. A slight increase in density was noted for laboratory mixed foam, 24.5 (1.53) to 26.6 kg/m^3 (1.66 PCF) compared with production machine mixed foam at 17.8 (1.11) to 20.2 kg/m^3 (1.26 PCF) and is attributed to a less thorough blending of ingredients.

2.2.4 Tensile Bond Strength using BX-251A-3D Foam

The tensile bond strength using BX-251A-3D foam core with Lefkowitz 211A/LZ-to-anodized 2219-T87 plates was not significantly different from strength data derived using BX-249N-3D foam core. The cryogenic bond strength was well within the expected range of 1.4 (200) to 2.1 MN/m^2 (300 psi). The ability of the bond to hold a load of 0.7 MN/m^2 (100 psi) at temperatures approaching 177°C (350°F) is also demonstrated by test data presented in later sections of this report.

The tensile bond strength of the glass fabric liner impregnated and bonded to BX-251A-3D foam also was within the normal range achieved using BX-249N-3D foam. The cryogenic bond strength of the liner is well above 1.0 MN/m^2 (150 psi) and the ability of the bond to hold a load of 0.7 MN/m^2 (100 psi) at temperatures over 149°C (300°F) was demonstrated. The liner 828/CL bond strength is customarily slightly lower than the tank wall 211A/LZ bond strength because of the resin content restriction to 60% by weight as explained in Section 2.3.1. It should be noted that liner bond strength to BX-251A-3D foam in which the 3D fibers were coated with silane primer, EC-3901, was more consistent than the strength of bond to 3D fibers without the silane treatment.

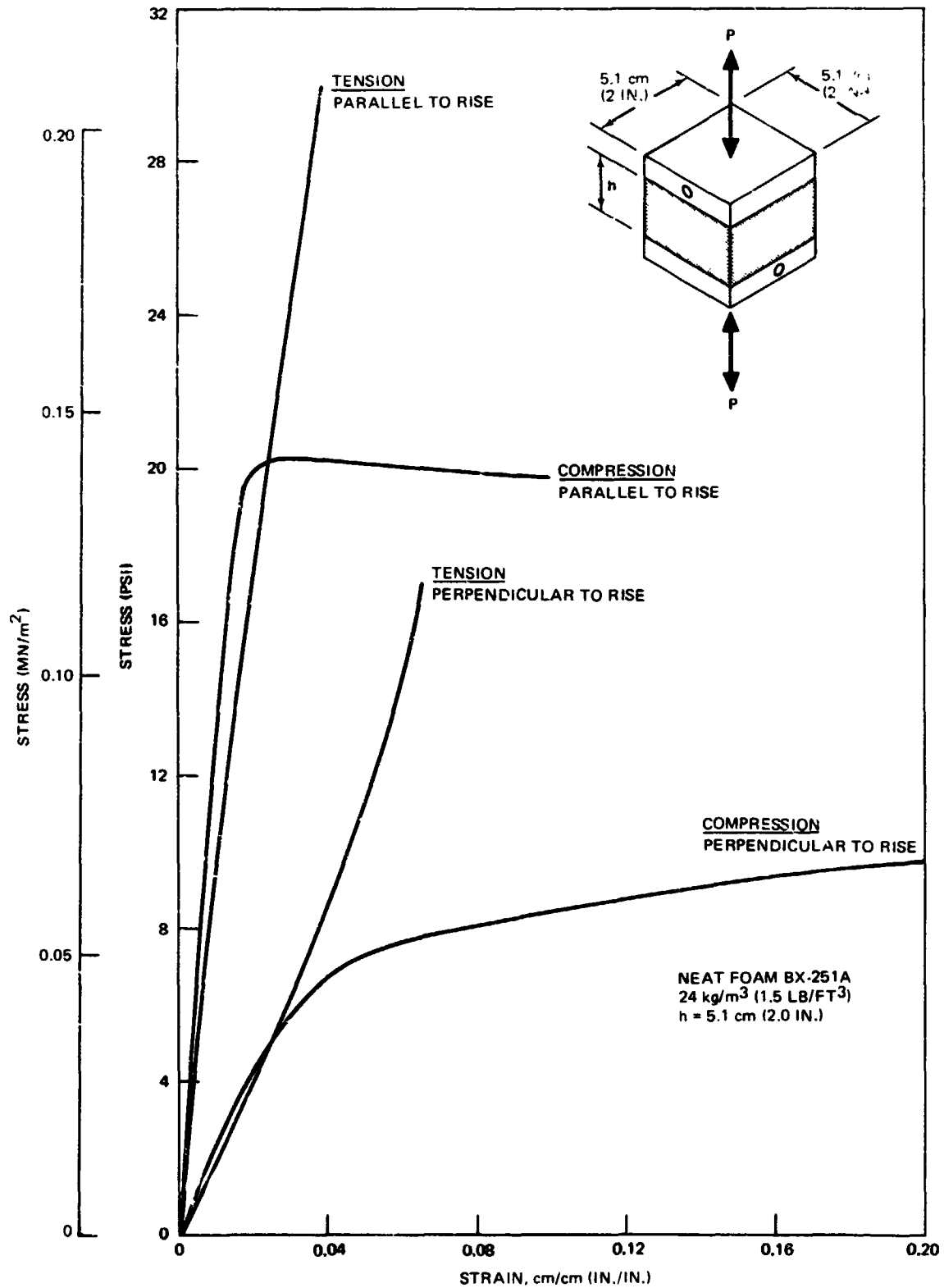


Figure 7. Stress-Strain Curves—Tension and Compression (Temperature 20°C) (68°F)

2.2.5 Compressive Strength of BX-251A-3D Foam (see Table 1)

As expected, the compressive strength of 3D blocks made with rigid epoxy resin pre-impregnated fibers, 0.68 MN/m^2 (98 psi), was higher than obtained with "as molded" blocks, 0.45 MN/m^2 (65 psi), in which only the foam was used to stiffen the 3D fiber array. Subsequent bonding operations simulating those performed during insulation of an LH_2 tank indicated that both 3D foam types possess the strength and abuse resistance to be successfully bonded to the tank wall under vacuum bag pressure and with personnel support during installation. Therefore, the increase in compressive strength provided by the additional process of fiber pre-impregnation was not considered to be worthy of the cost and weight increase. The option is available, however, to provide an increase in compressive strength should this be required for specific space vehicle applications.

2.2.6 Shear Strength (Plate) of BX-251A-3D Foam

The shear strength of 3D reinforced foam and unreinforced foam are presented in Figure 8, which illustrates the effect of fiber reinforcement on BX-251A polyurethane foam. The shear modulus of elasticity (G) of BX-251A foam was increased 100 percent by 3D fiber reinforcement, and the shear rupture strength was increased over 4 times by 3D fiber reinforcement.

2.2.7 Porosity of BX-251A-3D Foam

A judgment of porosity relative to 3D foam insulation was obtained using the test fixture described in Figure 9. A slice of 3D foam 2.5 cm (1.0 in.) thick was placed over an 18-cm (7.0-in.) diameter opening in a shallow chamber leading to a vacuum pump. With a metal plate substituted for the 3D foam slice, the vacuum gage connected to the shallow chamber will register 0.098 (29 in. Hg) within a few seconds. With a slice of Saturn S-IVB 3D foam, 83 kg/m^3 (5.2 PCF), placed over the opening the vacuum chamber gage will nominally register 0.051 MN/m^2 (15 in. Hg) with an established minimum acceptable value of 0.017 MN/m^2 (5 in. Hg). This test was devised to characterize the 3D foam core after the Saturn S-IVB subscale qualification tank tests proved the material was acceptable for flight operations, and the limits of acceptability were established using the test values measured on slices of 3D foam used to insulate the Saturn subscale tanks.

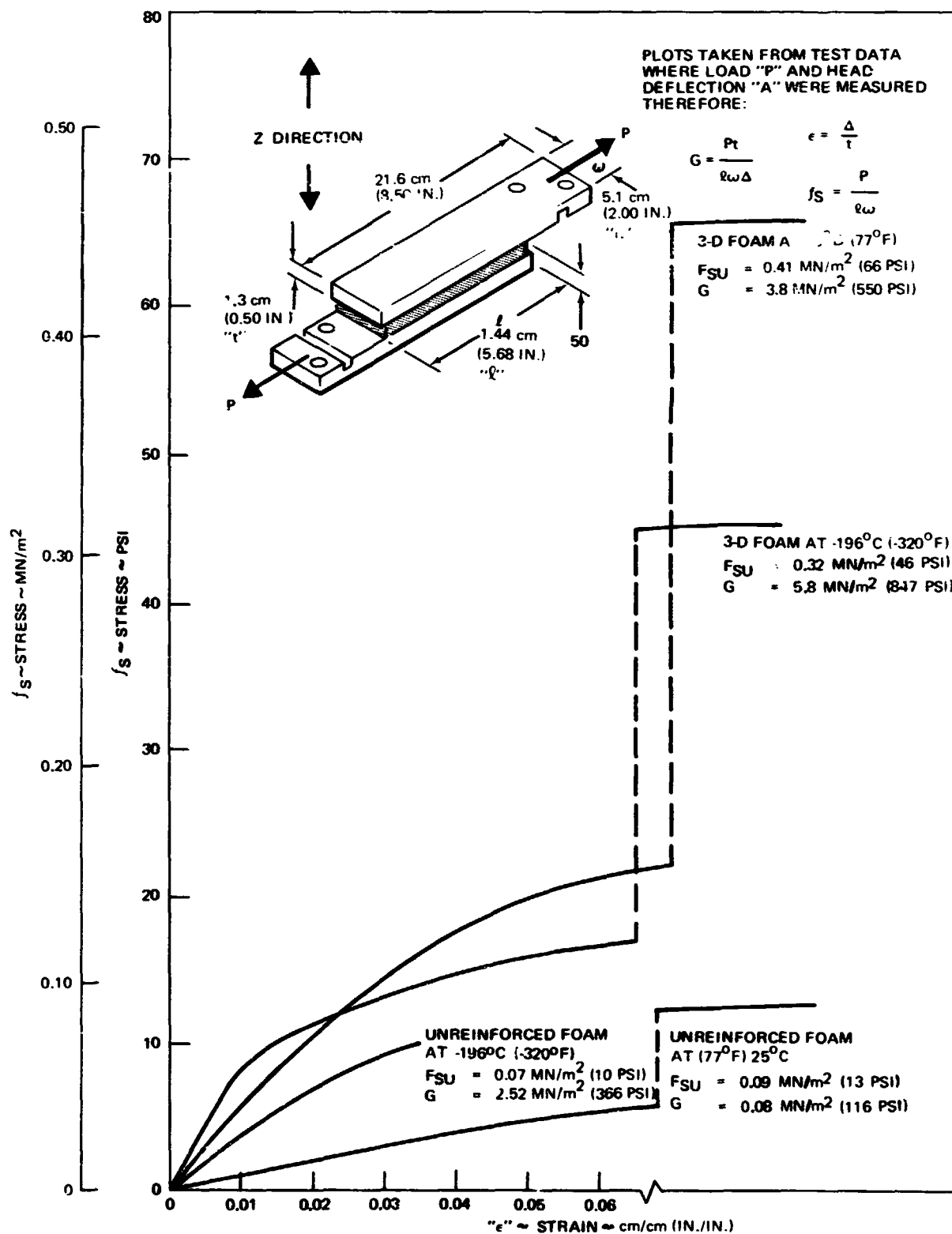
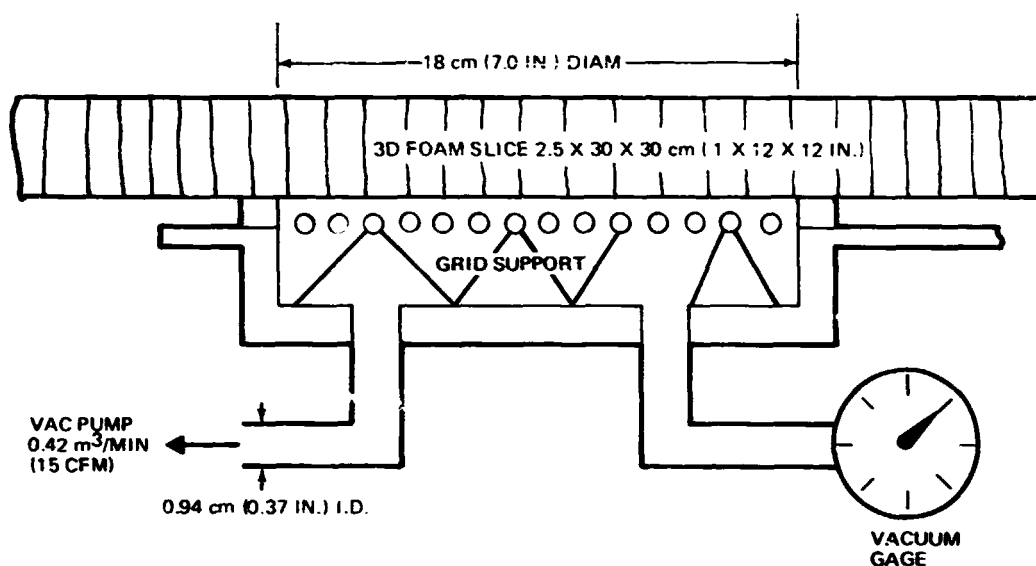


Figure 8. BX-251A Polyurethane Foam Shear Stress-Strain Curves (Z Direction)



- NOTE:
- WITH A METAL PLATE SUBSTITUTED FOR THE 3D FOAM SLICE, THE VACUUM GAGE WILL REGISTER 0.097 MN/m² (29 INCHES OF MERCURY) WITHIN 2 SECONDS
 - WITH A SLICE OF SIVB 3D FOAM, 83 kg/m³ (5.2 PCF), THE VACUUM GAGE WILL NOMINALLY REGISTER 0.051 MN/m² (15 IN. MERCURY) AND 0.017 MN/m² (5.0 IN. Hg) MINIMUM
 - ONLY THE SILANE PRIMER (EC3901) COATED FIBER BLOCKS OF BX251A, 43 kg/m³ (2.7 PCF), WOULD REGISTER ANY PRESSURE DIFFERENTIAL AND THESE SLICES VARIED BETWEEN 0.007 (2.0) AND 0.051 MN/m² (15.0 IN. Hg) WITH THE AVERAGE READING OF 0.020 MN/m² (6.0 IN. HG)

Figure 9. Porosity Test Fixture

In the case of BX-251A-3D foam slices tested for porosity, only the silane primer, EC-3901, coated fiber blocks offered sufficient resistance to air transfer to register a reading on the vacuum chamber gage. These silane coated fiber slices achieved a pressure differential gage reading of 0.007 (2.0) to 0.051 MN/m² (15.0) with an average of 0.020 MN/m² (6.0 inches of mercury).

This apparent advantage in low porosity and attendant improvement in fiber-to-foam bond afforded by the silane primer coating dictated the selection of this construction 3D foam for LH₂ tank insulation.

2.3 BONDING OPTIMIZATION (TASK 3)

Primary objective of this task was to determine the optimum mixing, application, and installation conditions for the tank wall adhesive. Allowable variations from optimum conditions were also determined.

A secondary objective of this task was to evaluate techniques for monitoring adhesive mixing, adhesive application, and insulation installation. This evaluation was designed to ensure that the insulation, as installed, would meet operational requirements.

As a result of Phase I research — baseline bonding processes for L211A/LZ and 828/CL were established. The L211A and LZ tank wall adhesive components were heated in separate containers at 82°C (180°F) for about 0.5 hours prior to mixing, and then thoroughly mixed by hand. The mixture ratio was 14 parts by weight LZ to 100 parts by weight L211A. When mixing the Epon 828 resin (used to impregnate and bond the 116 glass fabric liner to 3D foam) the required 12 parts by weight of CL hardener were melted at 82°C (180°F), to form an adduct. The hot adduct was then added to 88 parts by weight of Epon 828 resin at 25°C (77°F), resulting in a mixture ratio of 12 parts by weight of CL to 100 parts by weight 828. The mixture was then thoroughly mixed by hand. Fabric impregnation was accomplished within 15 minutes after mixing the resin.

The nominal baseline cure cycle, unless otherwise specified, was:

- A. Cure initially for 16 hours at $52 \pm 3^{\circ}\text{C}$ ($125 \pm 5^{\circ}\text{F}$) under vacuum-bag pressure, minimum of 20 in. Hg (51 cm Hg) and Shore A 90 minimum.
- B. Remove vacuum bag.
- C. Slowly heat the composite to reach 93°C (200°F) over a time period of not less than 3 hours, then raise the temperature to 150°C (300°F) over a time period of not less than 2 hours. A hardness above Shore A 90 was maintained at all temperatures.
- D. Postcure composite for 16 hours at 150°C (300°F).
- E. Cut composite into 5.1 cm (2 in.) by 5.1 cm (2 in.) blocks.
- F. Postcure (second time) for 16 hours at 150°C (300°F) to bond tensile blocks to composite blocks and to achieve additional strength in the foam-to-panel (or liner) composite block bond.

Throughout Phase I, the tensile bond test specimens were made using BX-249N-3D-S (BX-249 N foam, 3D reinforced, heat stabilized) and as-machined/anodized 2219-T87 aluminum alloy (unless otherwise noted). The liner specimens were made using BX-249N-3D-S (unless otherwise noted) and Epon 828/CL resin-impregnated 116 glass cloth with one rub coat. Liner specimens used one layer of 116-glass cloth, impregnated with 828/CL resin. Figure 10 illustrates the tensile test fixture used throughout this study for obtaining elevated temperature bond test data.

Figure 11 is a schematic of a typical LH_2 tank cure cycle. Figure 11 may be used to understand terminology employed in the bonding optimization program.

2.3.1 Adhesive Mixing Verification

The objective of this subtask was to evaluate methods for determining the thoroughness of the adhesive-catalyst mix, to establish the significant mixing parameters, and to specify requirements for a mixing machine for large-vehicle tankage use.

Variations in the thoroughness of mixing of the tank wall adhesive may have a significant effect on the reliability of the composite bond to the tank. In

TEST PROCEDURE:

1. ASSEMBLE SPECIMEN IN FIXTURE
2. POSITION HEATER ELEMENT PLATE FLAT AGAINST SPECIMEN BLOCK
3. LOAD IN TENSION TO 1,780 N (400 LB) 0.69 MN/m^2 (100 PSI)
4. ADJUST POWER TO HEATER TO REACH $\sim 204^\circ\text{C}$ ($\sim 400^\circ\text{F}$) ON THERMOCOUPLE INSIDE SPECIMEN BLOCK WITHIN 4 TO 5 MINUTES
5. RECORD TEMPERATURE AT RUPTURE

NOTE: DISCARD SPECIMENS THAT RUPTURE PRIOR TO APPLICATION OF HEAT OR THAT RUPTURE PRIOR TO TEMPERATURE INDICATION OF 93°C (200°F)

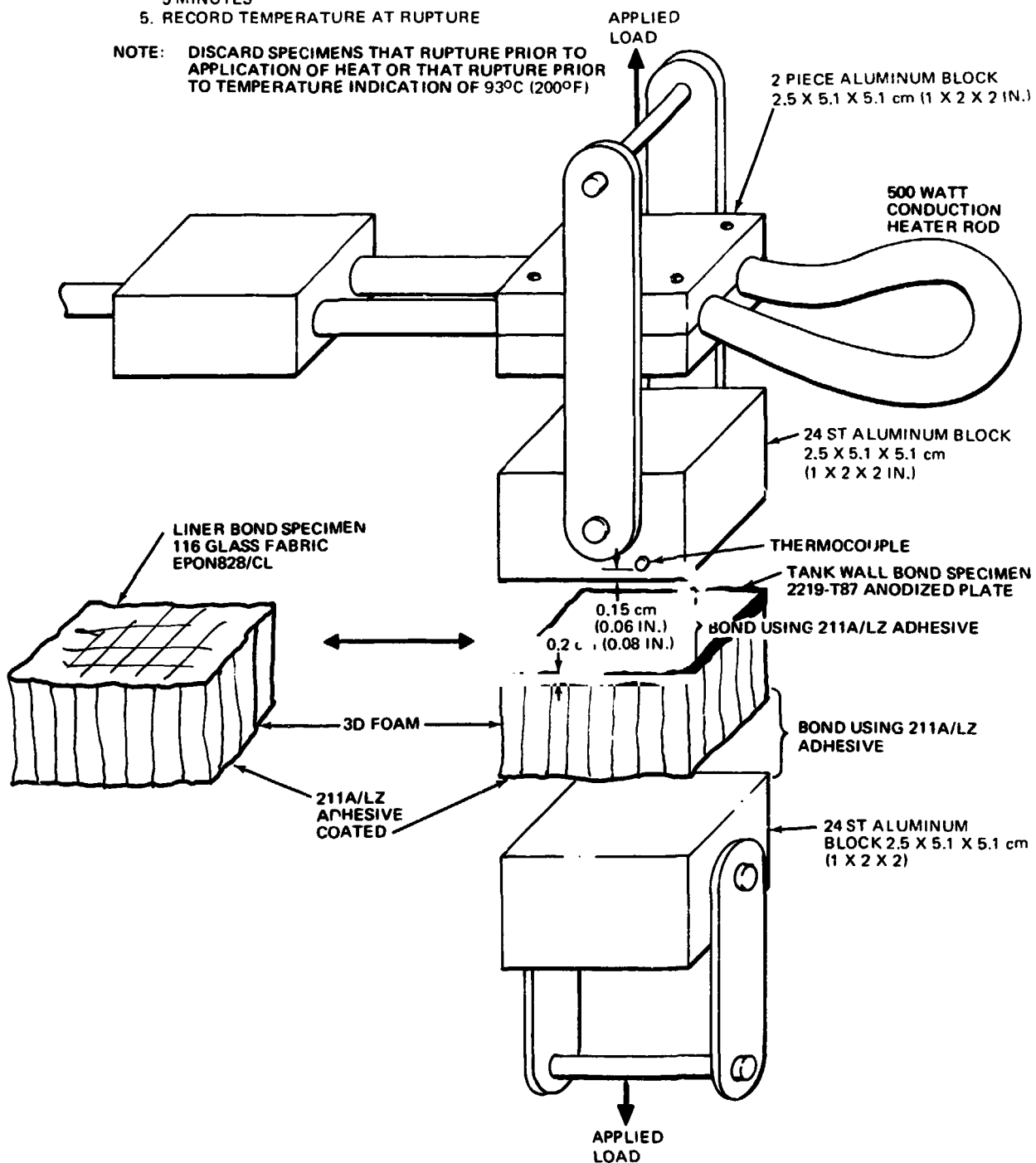
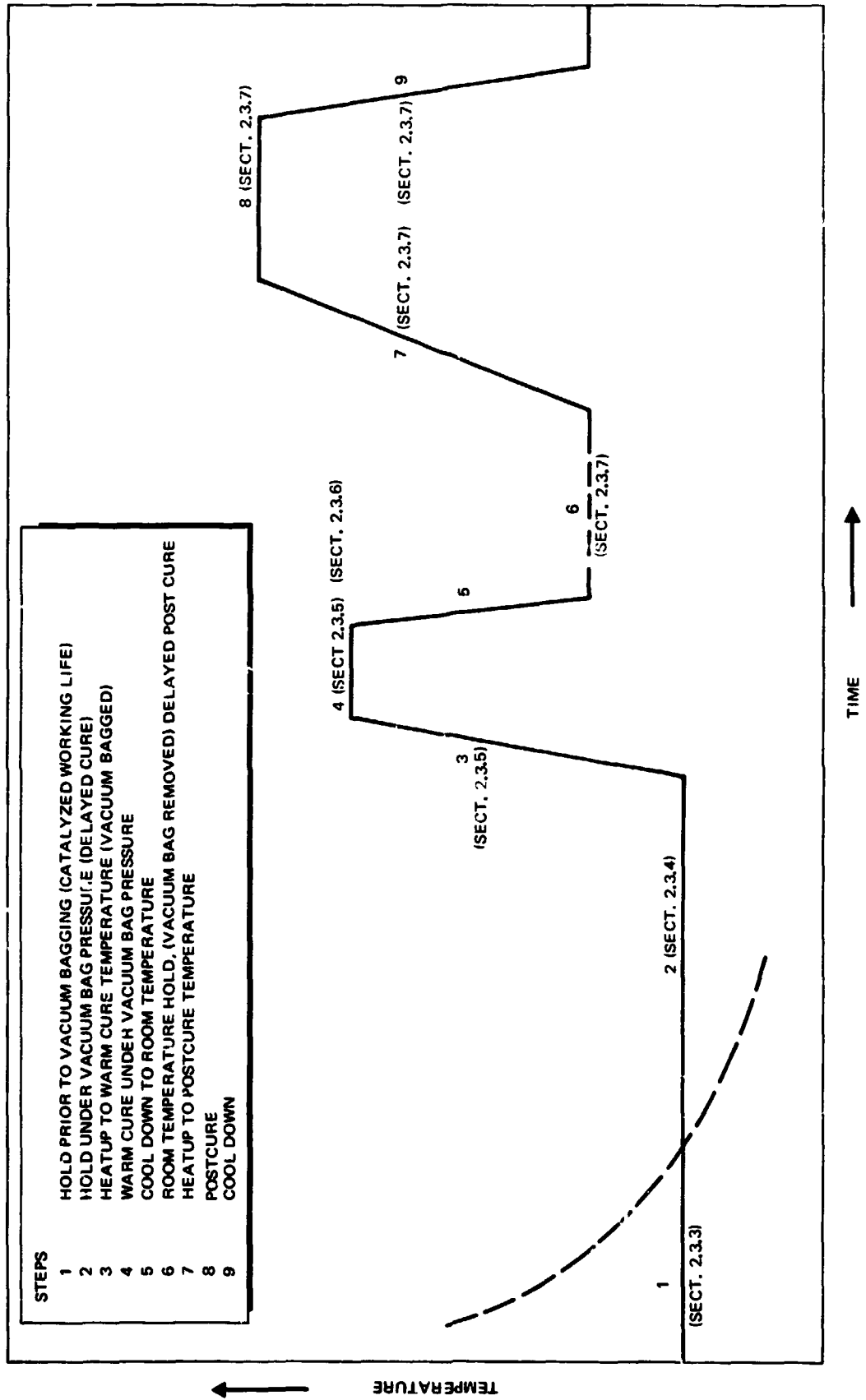


Figure 10. Elevated Temperature Test Fixture Tensile Bond Strength – Hot Plate

Figure 11. LH₂ Tank Cure Cycle

order to find a method for quickly and reliably identifying thoroughly mixed material, a survey was conducted. Techniques considered and tested to indicate feasibility for production included:

- A. Color change (dyes)
- B. Consistency (visual)
- C. Electrical impedance
- D. Tack and hardening time
- E. Fluorescence
- F. Thermogravimetric analysis (TGA)
- G. Attenuated total reflectance (ATR)

As a result of the survey, it was concluded that visual examination of mixed samples without dyes, using laboratory light, was the most suitable technique for determining thoroughness of mixing within 15 minutes after mixing, which is the time-scale selected for production monitoring. Results are summarized in Table 3.

Tests were conducted to obtain an indication of the effect of thoroughness of mixing and mixture ratio on the mechanical properties of tank wall adhesives. The results of these strength tests are presented in Tables 4 and 5. The data indicated that adhesive mixed to the point at which hardener streaks are barely visible (medium mix) still gives strengths about the same as those obtained on well-mixed material when tested at elevated and cryogenic temperatures. The same is true of material with a 10 percent high or 10 percent low hardener-to-resin mixture ratio. These data were used to determine whether the degree of mixing and mixture ratio of the L211A/LZ mixing machine (discussed later) were adequate.

Adhesive having visual streaks of hardener component can develop a measurable hardness (by Shore Durometer) more rapidly than well-mixed adhesive, but retains soft tacky spots on the surface and within the thickness that indicate an abnormal mix condition. Although bond strength specimens did not show abnormal bond-strength values, the potential exists for other properties to be affected by streaked, improperly mixed adhesive and on this basis no test panels, test tanks, or production vehicles will be bonded using adhesive in which hardener streaks are visible.

Table 3
EVALUATION OF METHODS FOR DETERMINING ADHESIVE MIX THOROUGHNESS

Method	Technique	Time Required (min)	Reliability
A. Color change	Visual—dyes	~15	High
B. Consistency examination	Visual	~15	High
C. Electrical impedance	Ac phase angle and reactance; dc resistance	~30	Moderate (additional work required)
D. Tack	Mechanical—manual	>60	Moderate
E. Fluorescence	Visual—black light	~20	Moderate
F. Thermogravimetric	Weight loss vs. temperature	>60	Low
G. Attenuated Total Reflectance (ATR)	Infrared absorption spectra	>30	High

Table 4
DEGREE OF ADHESIVE MIXING

Degree of Mixing	Tensile Strength at -196°C (-320°F)		Temperature at Rupture Under Stress of 0.7 MN/m ² (100 psi)	
	MN/m ²	(psi)	°C	(°F)
Poor (0.5 min)	0.181	(262)	149	(300)
	0.152	(220)	149	(300)
	0.216	(313)	134	(273)
	Average 0.182	(264)	Average 144	(291)
Medium (1 min)	0.200	(290)	163	(326)
	0.153	(222)	160	(320)
	0.145	(211)	171	(340)
	Average 0.167	(242)	Average 165	(329)
Moderately well (3 min)	0.169	(245)	161	(322)
	0.216	(313)	170	(338)
	0.224	(325)	163	(326)
	Average 0.204	(295)	Average 165	(329)
Nominally well (10 min)*	~0.17	(250)	~170	(340)

*Of the order of a total of 200 specimens

The emphasis placed on production operational methods encompassed by the intent of this development program dictated the need to qualify a mechanical means to meter the adhesive ingredients, blend thoroughly, and dispense the mixed adhesive uniformly over the 3-D foam surface to obtain coverage of approximately 0.48 kg/m² (45 grams/ft²). The experience gained from bonding 3-D foam tile in the LH₂ tanks of the Saturn S-IVB using a mechanical mixing and dispensing unit was used to establish the requirements for a

Table 5
MIXTURE RATIO VARIATION

Variation of Hardener from Nominal 14-pbw LZ/100-pbwa L211A Mixture Ratio	Tensile Strength at -196° C (-320° F)		Temperature at Rupture Under Stress of	
	MN/m ²	(psi)	0.7 MN/m ² ° C	(100 psi) (° F)
+10%	0.75	(109)	170	(338)
	0.172 ^b	(250)	179	(355)
	0.204 ^b	(295)	174	(346)
	Average 0.150 ^c	(218)	174	(346)
-10%	0.89	(129)	177	(350)
	0.110 ^b	(160)	171	(340)
	0.190 ^b	(275)	174	(345)
Nominal*	Average 0.130 ^c	(188)	170	(346)
	~0.17	(250)	~170	(340)

^a pbw means parts by weight.

^b Indicates specimen ruptured primarily against grit-blasted tensile block or between tensile block and metal plate.

^c "greater than"

*Based on of the order of 200 specimens.

new unit capable of mixing and dispensing the high-viscosity Lefkowitz 211A LZ adhesive for this program. A survey of available commercial equipment was conducted which resulted in the selection of SEMCO (Glendale, Calif) to develop a mixing machine capable of handling Lefkowitz 211A/LZ adhesive. A successful demonstration was performed by SEMCO using a Model 1384 high-pressure pump assembly with a Model 1383 blending head. This unit was subsequently procured independently by MDAC and used in the fabrication of bonded panels tested during the latter part of this program.

Figures 12 and 13 show this mixing machine in operation during a simulated production sequence of continuously coating 3-D foam tile with Lefkowitz 211A/LZ adhesive.

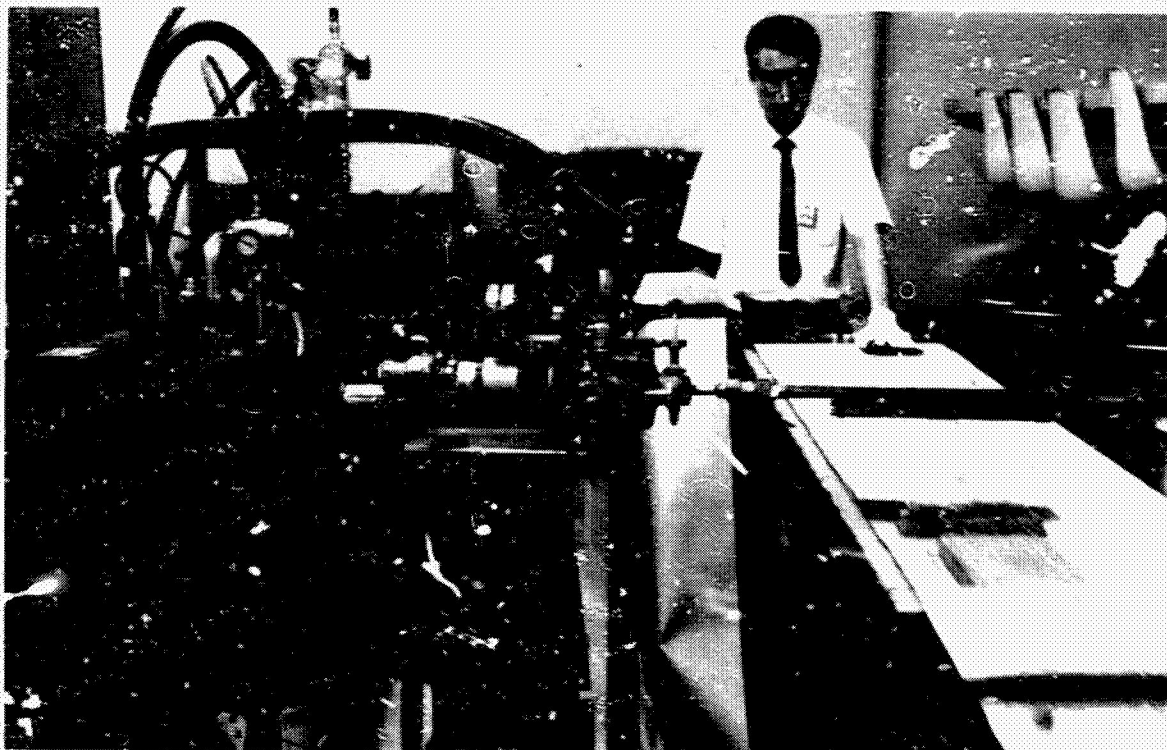


Figure 12. Tank Wall Adhesive Mixing Machine

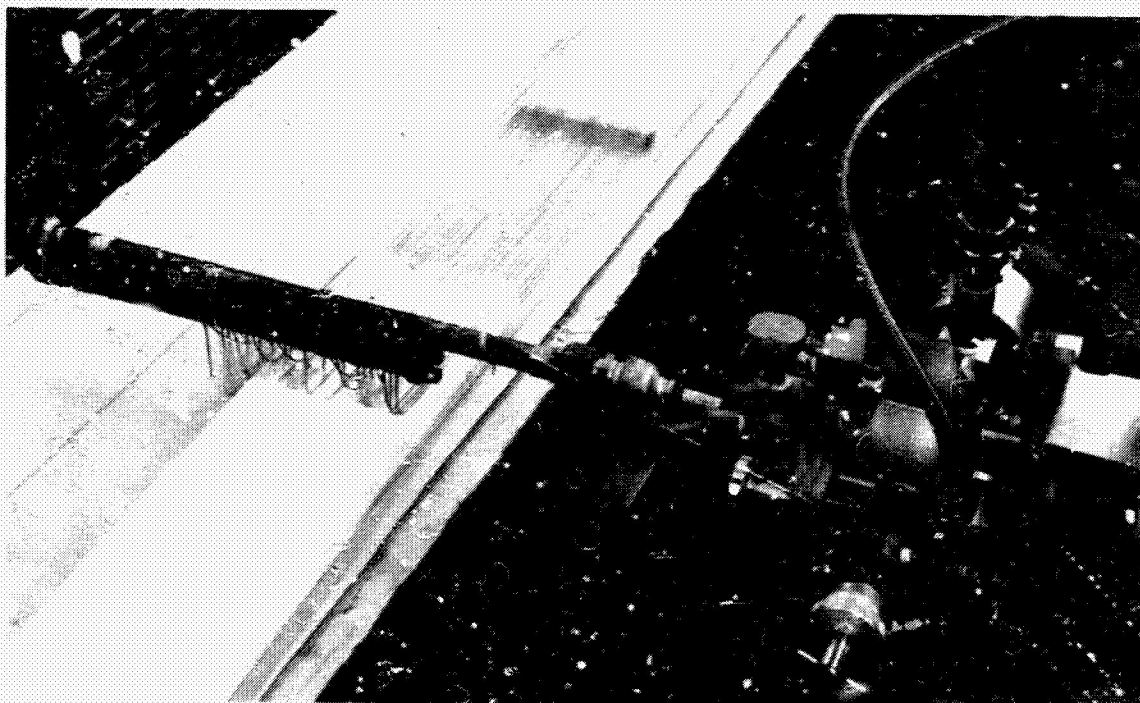


Figure 13. Tank Wall Adhesive Extruder

The SEMCO mixing machine proved to be capable of depositing Lefkowied 211A/LZ adhesive on the 3-D foam surface at rates between 0.37 (4) and 1.85 m²/min (20 ft²/min). Deposition can be adjusted between 0.21 (20) and 0.97 kg/m² (90 grams/ft²) within a tolerance of ± 20 percent. The rate of coverage per square meter (foot) should be equal to or greater than 0.74 m²/min (8 ft²/min) in order to keep pace with 3-D form tile laying operations.

The bond strength of machine-mixed Lefkowied 211A/LZ was determined and the results are presented in Table 6, showing bond strength equal to or greater than that achieved with hand-mixed adhesive. The catalyzed working life of machine-mixed Lefkowied 211A/LZ was generally longer than obtained using hand-mixed adhesive and offered a distinct advantage in this regard; however, the catalyzed working life curve presented later in Section 2.3.2 does not reflect this advantage and as such is considered to be conservative.

2.3.2 Minimum Weight Adhesive for Bonding Foam to Tank Wall

The objective of this subtask was to determine the minimum tolerance on Lefkowied L211A/LZ adhesive weight that can be used to reliably bond the 3-D foam to the tank wall.

The foundation of this effort was derived from experiments in which the entire surface of the 3-D foam was coated with adhesive, per the baseline, at 0.48 (45) to 0.54 kg/m² (50 grams/ft²). Cellophane film was then pressed against the adhesive layer and peeled off, taking the adhesive off the foam surface, but leaving adhesive on the thread ends. The fact that Lefkowied 211A/LZ will adhere to a continuous smooth surface, presented by the cellophane film, with greater tenacity than to the cut edges of the foam cells was used to advantage in this attempt to deposit (or leave) adhesive only on the Z fiber ends. The tufted ends of the Z fibers tend to hold the adhesive in place without transferring to the cellophane film. Figure 14 shows the surface coverage using the nominal 0.48 kg/m² (45 gm/ft²). Figure 15 shows the surface coverage using 0.34 kg/m² (32 gm/ft²). Figure 16 shows the surface coverage using 0.19 kg/m² (18 gm/ft²). In each case, the Z fiber fillet appears to be well defined. Acceptable bond strength was obtained

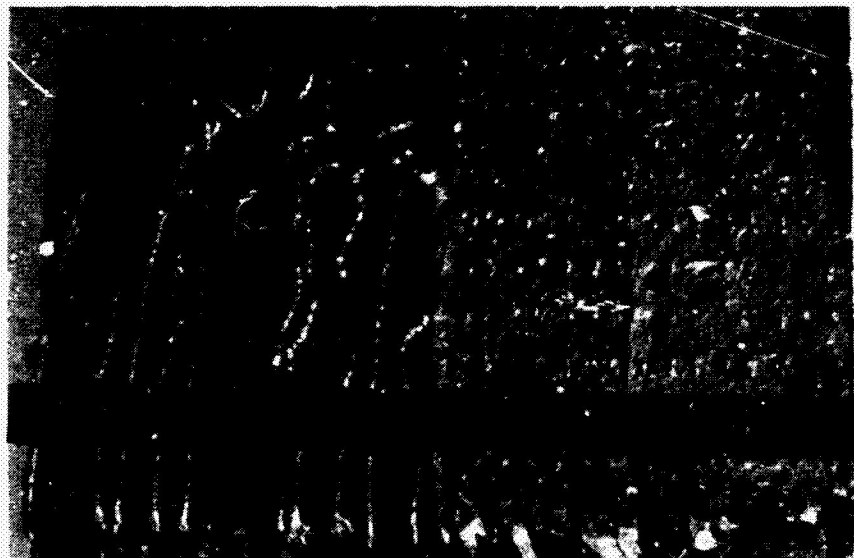
Table 6
MACHINE-MIXED ADHESIVE^a

Mixing Head Rotor Speed (rpm)	Tensile Strength at -196° C (-320° F)		Temperature at Rupture 0.7 MN/m ² (100 psi)	
	MN/m ²	(psi)	° C	(° F)
250 (nominal)	0.165	(240)	163	(325)
	0.125	(182)	163	(325)
	0.128	(186)	163	(325)
	Average 0.139	(202)	Average 163	(325)
	0.190	(275)	144 ^b	(290)
450 (maximum)	0.206	(299)	166	(330)
	0.116	(168)	181	(358)
	Average 0.171	(248)	Average ~170	(338)

^aSpecimens were bonded after 17 hours at 25° C (77° F). The machine dispensed the material at 38.2 l/hr (9.54 gal/hr).

^bSpecimen ruptured primarily against grit-blasted tensile block or between tensile block and the metal plate.

CR83



AS EXTRUDED
0.18 cm (0.07 IN.) DIA
X 0.63 cm (0.25 IN.)

SCRAPE SMOOTH
ONE PASS WITH
BLADE AT 82° C
(180° F)

Figure 14. Lefkowied 211A/LZ, 0.48 kg/n.² (45 Gm/Ft²)



Figure 15. Plexiglas Plate Adhesive Flow Test



Figure 16. Plexiglas Plate Adhesive Flow Test

at 177°C (350°F) and at -196°C (-320°F) with only 0.19 kg/m² (18 gm/ft²) of Lefkowitz L211A/LZ adhesive on the thread ends, as presented in Table 7. These experiments were conducted only to assess the structural feasibility of using adhesive at the Z thread fillet contact points and to judge the amount of adhesive weight that could be removed from the foam surface. The development of mechanical equipment necessary to accomplish this type of deposition was not considered compatible with the intent of this program, since it would unnecessarily delay the investigations to determine adhesive working life and optimum curing conditions. This option of low adhesive weight is available, however, and can be activated downstream and when the cost-effectiveness parameters are more clearly established.

The SEMCO mixing machine has positive-displacement metering cylinders that deliver the ingredients to the blender in a ratio measured at 13.4 parts of LZ hardener to 100 parts 211A adhesive. Previous strength tests with hand-mixed adhesive having a low of 12.6 parts LZ and a high of 15.4 parts LZ indicated no significant difference when compared with the nominally mixed adhesive using 14.0 parts LZ. The strength tests conducted on adhesive mixed by the machine delivering 13.4 parts LZ and reference panels of hand-mixed 13.4 parts LZ developed equivalent strength values as anticipated. Therefore, the metering cylinders on the machine were not altered and the ratio of 13.4 parts LZ to 100 parts 211A was considered to be acceptable.

The temperature at which the ingredients are stabilized or stored prior to machine blending proved to be of crucial importance. The LZ hardener will crystallize at temperatures below 21°C (70°F) and must be heated to 82°C (180°F) to 94°C (200°F) for 0.5 hour to remelt the crystals, and thereafter maintained in a temperature-controlled chamber at 27°C (80°F) to 32°C (90°F). In effect, this means the material storage area and the entire mixing machine must be maintained at 27°C (80°F) to 32°C (90°F) in order to maintain a state of readiness for bonding operations.

2.3.2.1 Epon 828/CL Resin Mixing and Optimum Weight in Glass Liner

The baseline resin system for impregnating and bonding the 116 glass fabric liner to 3-D foam is Epon 828 with metaphenylene diamine (CL) hardener in a nominal ratio of 100 parts resin to 12 parts CL hardener. This resin is

Table 7
BOND STRENGTH USING LOW-WEIGHT 211A/LZ
ADHESIVE APPLICATION

Application Techniques	Adhesive Coating Weight		Tensile Strength -196°C (-320°F)		Temperature at Rupture under Stress of	
	(g/m ²)	(g/ft ²)	(kg/cm ²)	(psi)	7.0 kg/cm ² (°C)	100 psi (°F)
Manual application to foam and metal	537	50	20.4*	291	174	346
			12.9*	184	178	352
			14.4	205		
			Average		Average	
			>15.9	227	176	349
Extrusion on foam only, from holes spaced 0.31 cm (0.12 in.) on center	980	91	11.2*	160	177	350
			11.2	159	168	335
			16.6	237	116	330
			18.0*	256		
			Average		Average	
Extrusion on foam only, from holes spaced 0.31 cm (0.12 in.) on center	484	45	15.3*	218	163	325
			12.3*	175	163	325
			15.7*	224	167	332
			15.1	215		
			Average		Average	
Plus blotting (cellophane transfer)	193	18	>14.6	208	164	327
			14.3	204	171	340
			14.7	210	168	335
			16.5	235	118	245
			Average		Average	
Plus blotting (cellophane transfer)	75	7	15.2	216	153	307
			11.6	166	5.5 kg/cm ²	(78 psi)
			6.0	86		
			12.3	176	5.5 kg/cm ²	(78 psi)
			8.7	124	7.0 kg/cm ²	(100 psi)
			Average			
			9.7	138		

NOTE: Following specimens ruptured prior to application of heat; only one reached 100-psi loading

*Specimen failed primarily against grit-blasted tensile block or between tensile block and metal plate

unfilled and will transmit light. The index of refraction of the mixed resin was used to determine thoroughness of blend and also to verify the correct ingredient ratio. A description of the refractometer is given in Figure 17 and the graph for determining the correction factor for temperature changes is presented in Figure 18. The mix ratio tolerances for Epon 828/CL are given in percent by weight of CL to Epon 828 and can vary between 10.2 and 11.7 percent. The corresponding refraction index reading of 1.5713 to 1.5719 at 25°C was used to control the thoroughness of mix and mix ratio.

The minimum resin content used in bonding the liner to 3D foam was dictated by the bond strength requirement of at least 0.69 kg/m^2 (100 psi). A review of all liner bond strength values determined in this program with Epon 828/CL will indicate the liner bond was slightly weaker than the tank wall bond using 211A/LZ adhesive. Therefore, no serious attempt was made to decrease the resin content below 60 percent (or 0.16 kg/m^2 ; 15 gm/ft^2).

Larger resin fillets at the Z fiber contact points would result in raising the bond strength but would also raise the contraction stress in the liner when exposed to LH_2 . The margin between the contraction stress and the rupture strength of the liner when tested at -253°C (-423°F) (LH_2) is presented in Table 8. Sixty percent resin content is very close to that obtained with the S-IVB liner, which had an excellent history of resistance to rupture in LH_2 tanks. In addition to the contraction stress in LH_2 , the liner must withstand the stresses induced by the expanding tank wall during the pressurization period of engine firing. For these reasons, the resin content was not increased in the Epon 828/CL impregnated glass liner.

2.3.3 Catalyzed Working Life

The objective of this subtask was to determine the allowable span of time between adhesive application and the application of vacuum bag pressure, and the dependence of this time on the tank wall temperature.

The Saturn S-IVB adhesive system is applied to the 3-D foam; the foam is placed against a cold (13°C ; 55°F) tank wall, and allowed to stay in place without pressure for approximately 5 hours. This procedure allows a large area of the tank to be insulated prior to the application of vacuum bag

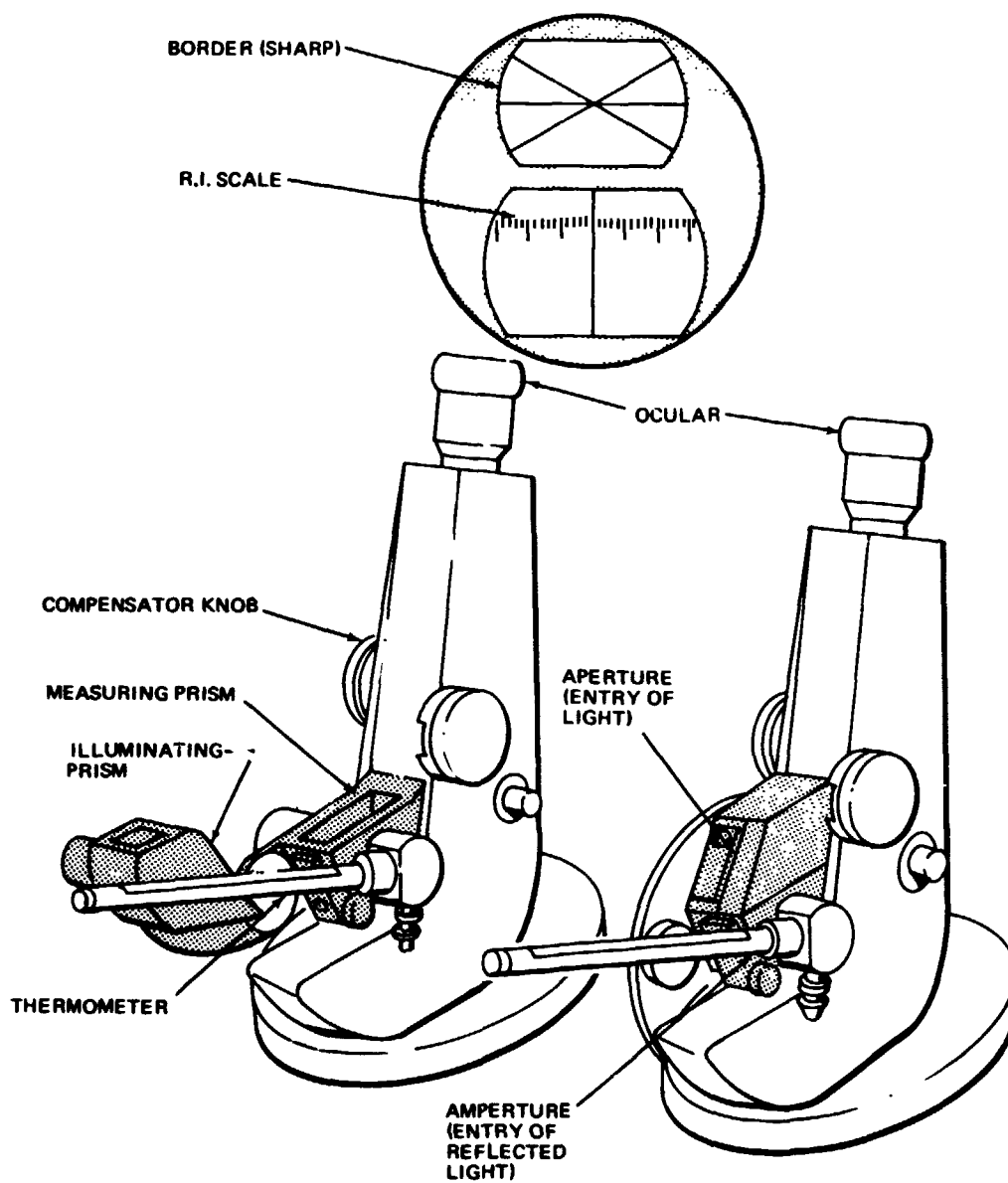


Figure 17. Zeiss Opton Abbe Refractometer

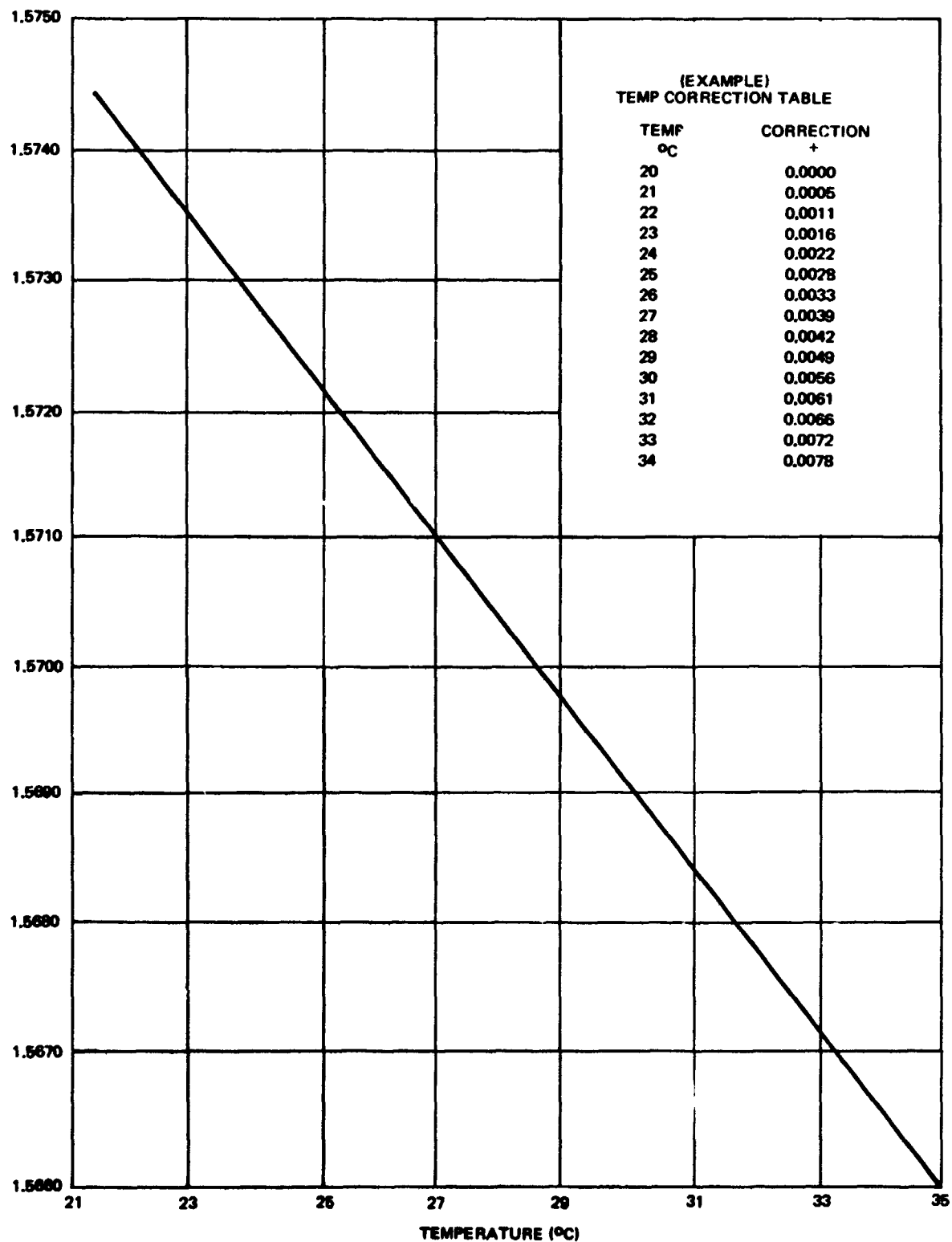


Figure 18. Index of Refraction – Temperature Correction Graph for EPON 828/CL Resin

Table 8
LINER CONTRACTION TESTS

Liner Type	Contraction Load		Ultimate Tensile		Ratio of Contraction Load to Ultimate Tensile Load
	N/m width	(lb/in. width)	N/m width	(lb/in. width)	
S-IVB	3.40	(194)	8.05	(460)	0.422
	4.41	(252)	7.80	(446)	0.565
	4.52	(258)	8.40	(480)	0.538
	3.68	(210)	7.43	(424)	0.497
Average	3.99	(228)	7.90	(452)	0.505
Shuttle Liner (Phase II baseline)	2.80	(160)	6.20	(354)	0.452
	3.05	(174)	5.92	(338)	0.515
	2.52	(144)	5.99	(342)	0.421
	2.45	(140)	5.77	(330)	0.425
Average	2.70	(154)	5.97	(341)	0.452

pressure. The length of time the adhesive can remain at this cold temperature, and subsequently develop good flow properties, determines the surface area that can be insulated in one adhesive operation. If the allowable hold time before initiating the warm cure cycle is sufficiently long (on the order of 70 hours), an entire LH₂ tank may be insulated in one operation, with a consequent reduction in total installation time and cost.

For these tests Lefkowitz 211A/LZ was spread over the surface of a slice of 3D foam to achieve 0.48 kg/in.² (45 gm/ft²) immediately after mixing in a manner closely simulating the automatic mixing and dispensing system anticipated for production operations.

The coated 3D foam and mating aluminum plate were both placed in a temperature conditioned chamber. In production, the coated 3D foam tile would be positioned against the aluminum tank wall and the adhesive would be in partial contact with the aluminum until vacuum bag pressure was applied. Since these tests were intended to represent the most severe condition, no contact was allowed between adhesive and aluminum plate during the period prior to vacuum bag pressure application. Also, the coated 3D foam and aluminum plate were not covered and were exposed to internal "tank" environment. Hardness specimens of adhesive 0.30 cm (0.12 inches) thick were put

in polyethylene bags, placed adjacent to each panel, and monitored by Shore durometer readings throughout the sequence.

The holding time period was estimated by examining the hardness specimens, The first panel at each temperature condition to receive vacuum bag pressure was considered to be almost as flowable as using freshly mixed adhesive.

The succeeding panels to receive vacuum bag pressure were spaced at time intervals estimated so that the last panel would be incapable of achieving a flow pattern under vacuum bag pressure. The last panel to be pressured, and generally the previous panel or two, did not bond and separated after the cure cycle as though the metal plate were coated with parting agent.

All panels for these tests were enclosed in a vacuum bag while still under "tank" temperature conditions and pressured to 0.068 MN/m^2 (20 in. Hg). The cure cycle was carried out at 52°C (125°F) for a standard time of 16 hours. The vacuum bag was removed and the panel slowly heated to 149°C (300°F) over a two hour period then held at 149°C (300°F) for a standard time of 16 hours.

A group of 10 specimens, $5.1 \times 5.1 \text{ cm}$ (2 x 2 inches), were machined from each panel. Six of these specimens were prepared for bonding to the aluminum blocks used in testing fixtures for measuring tensile bond strength by lightly grit blasting the mating surfaces. The adhesive used for specimen mounting was freshly mixed Lefkowitz 211A/LZ. Vacuum bag pressure was again applied to bond the mounted specimens and cured directly at 149°C (300°F) for another 16 hours. The adhesive bond line under test was therefore exposed to a total cure time of 32 hours at 149°C (300°F).

Three specimens from each panel were given the tensile bond strength test at 196°C (-320°F) (LN_2 soak) and three specimens were loaded to 1,780 N (400 pounds) (0.69 MN/m^2 ; 100 psi) and heated using a hot plate until bond line rupture occurred.

Over 100 panels were prepared during this series of tests and over 300 specimens were prepared for strength evaluation at cryogenic and elevated temperatures.

The guidelines for acceptance relative to bond strength of specimens subjected to these processing variables were based on strength values achieved by "reference" specimens made with freshly mixed 211A/LZ adhesive, or Epon 828/CL liner, and processed under laboratory conditions. These strength guidelines included:

- A. Tensile bond strength immersed in LN_2 (-196°C ; -320°F) of:
0.69 MN/m^2 (100 psi) minimum
1.1 MN/m^2 (150 psi) average
and should be in the same range of strength as obtained by testing "reference" specimens.
- B. While holding a constant tensile load of 0.69 MN/m^2 (100 psi) the bond line shall be capable of being heated to over 177°C (350°F) before rupturing, and should rupture within or above the temperature range achieved by testing reference specimens.

2.3.3.1 Effect of Prolonged Catalyzed Working Life on L211A/LZ

The maximum hold temperature selected for examination was 52°C (125°F) [all temperatures fluctuated as much as $\pm 3^\circ\text{C}$ ($\pm 5^\circ\text{F}$) during exposure]. Obviously no significant area of production bonding would take place at 52°C (125°F). However, repair operations could be required during the initial cure cycle while the tank is at this temperature. The first panel pressured after 2.5 hours exhibited marginal strength at high and low test temperatures indicating too long a hold period at 52°C (125°F). The next panel pressured after 3.0 hours developed higher strength values and in the range expected with freshly mixed adhesive. The third panel pressured after 4.0 hours did not bond together even though the adhesive flowed completely over the metal surface when pressure was applied. The remaining panels were discarded. Hardness measurements indicated that no bond could be expected after reaching Shore durometer A30 at 52°C (125°F). Allowing for variation in adhesive formulation from batch to batch, a safe hold time was established for 52°C (125°F) at 1 hour.

The next "tank" temperature to be examined was 43°C (110°F). The first panel was pressured after 4.0 hours and again exhibited marginal strength; however, the panels pressured after holding periods of 6.5 and 7.5 hours developed acceptable strength values indicating the possibility of an irregularity in the manner of specimen preparation in the testing of the first

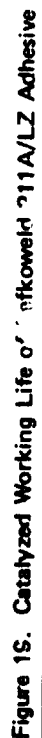
(4.0 hour) panel. The fourth panel held at 43°C (110°F) for 8 hours prior to pressure application did not bond together and separated easily after the cure cycle. The remaining panels were discarded. Hardness measurements indicated that no bond could be expected after reaching Shore durometer A25 at 43°C (110°F). Allowing for variation in Lefkowitz 211A/LZ adhesive formulation from batch to batch, a safe hold time was established for 43°C (110°F) at 6.0 hours.

At 25°C (77°F) hold temperature the panels were pressured at 7.0 hours, 17.0 hours, 21 hours, 24 hours and 30 hours. The bond strength held normal through 21 hours holding period and dropped to zero on the 24 hour and 30 hour panels, which were discarded. Hardness measurements indicated that no bond could be expected after reaching Shore A50 at 25°C (77°F). The hardness dropped to Shore A20 when warmed to 52°C (125°F) cure temperature. Allowing for batch to batch variation in adhesive formulation a safe hold time was established for 25°C (77°F) at 17 hours.

The same procedure as described above was used to determine the hold time at 2°C (25°F) then additional panels were tested at 13°C (55°F) in order to refine the curve showing the safe hold time and temperature along with adhesive hardness measurements.

Hardness measurements provided an indication of acceptable flow and bondability that can be used during manufacturing operations, but at temperatures below 18°C (65°F) the hardness reading was affected more by freezing than by polymerization of the adhesive. It would be hazardous to depend on hardness readings taken at 2°C (35°F) because of the drastic change with slight temperature fluctuations. The cold specimens must be warmed to at least 25°C (77°F) prior to taking the hardness measurement. At 25°C (77°F) the safe maximum hardness was established as Shore A40 prior to the application of vacuum bag pressure.

The working life curve showing safe limits of catalyzed working life along with the Shore hardness measurements is shown in Figure 19.



2.3.3.2 Effect of Prolonged Catalyzed Working Life on 828/CL Liner, Impregnated 116 Glass Fabric

For these tests, the resin Epon 828/CL was spread over the 116 glass fabric to achieve $0.16 \pm 0.02 \text{ kg/m}^2$ ($15 \pm 2 \text{ gm/ft}^2$) of resin.

Dry 116 Glass Fabric = 0.11 kg/m^2 (10 gm/ft^2)

Epon 828/CL Resin = 0.16 kg/m^2 (15 gm/ft^2)

Total = 0.27 kg/m^2 (25 gm/ft^2)

A carrier film of 0.1 mm (6 mil) perforated polyethylene was used to allow handling without wrinkling the impregnated fabric. Impregnation of the fabric was accomplished within 15 minutes after mixing the hardener and resin together.

The impregnated fabric and mating slice of 3D foam were both placed in a temperature conditioned chamber. In production, the impregnated fabric would be positioned directly against the 3D foam already in place against the tank wall and the Z fiber ends of the 3D foam would receive a partial coating of wet resin transferred from the impregnated fabric. These tests were intended to represent the most severe condition, therefore, no contact was allowed between the impregnated fabric and 3D foam during the period prior to vacuum bag pressure application. Also, the fabric and 3D foam were not covered and were exposed to internal tank environment. Hardness specimens of Epon 828/CL resin, 0.30 cm (0.12 in.) thick were poured into polyethylene bags and were placed adjacent to each panel and were monitored by Shore durometer reading throughout the sequence.

In addition to hardness specimens of Epon 828/CL an indication of the ability of this resin to wet the Z fibers of the 3D foam was achieved through the use of an acrylic plate 0.15 x 3.8 x 13 cm (0.06 x 1.5 x 5.0 in.) having dry glass fibers taped across the surface. At the same time the impregnated fabric was placed against the mating 3D foam, this acrylic plate was inserted under the excess edge of the fabric exposing the dry glass threads on the plate to the resin within the fabric. Vacuum bag pressure of 0.068 MN/m^2 (20 in. Hg) was then applied, and the assembly was cured for the standard cure cycle of 16 hours at 52°C (125°F). Examination of the glass threads on the acrylic plate by 10X magnification revealed either complete resin impregnation,

partial impregnation or no impregnation. These notations were then correlated with the tensile bond strength test results conducted at high and low temperatures. A photograph of the acrylic plate and threads typical of this flow test is presented in Figure 20.

The holding time period was estimated by examining the hardness specimens in identical manner as previously described for Lefkoweld 211A/LZ.

All panels for these tests were enclosed in a vacuum bag while still under "tank" temperature conditions and pressured to 0.068 MN/m² (20 in. Hg). The cure cycle was carried out at 52 °C (125 °F) for a standard time of 16 hours. The vacuum bag and perforated polyethylene carrier film were removed and the panel slowly heated to 149 °C (300 °F) over a 5 hour period then held at 149 °C (300 °F) for a standard time of 16 hours. The specimen preparation procedure, including another cure cycle at 149 °C (300 °F) for 16 hours, was the same as that previously described for Lefkoweld 211A/LZ adhesive.

Three specimens from each panel were given the tensile bond strength test at -196 °C (-320 °F) (LN₂ soak) and three specimens were loaded to 1,780 N (400 pounds) (0.69 MN/m², or 100 psi) and heated using a hot plate until bond line rupture occurred between liner and 3D foam.

Over 100 panels were prepared during this series of tests and over 200 specimens were prepared for strength evaluation at cryogenic and elevation temperatures.

2.3.3.3 Effect on Epon 828/CL Liner Bond After Prolonged Holding Time at Various Temperature Prior to Application of Vacuum Pressure

At a hold temperature of 52 °C (125 °F) the Epon 828/CL exhibited a more sensitive, faster cure than Lefkoweld 211A/LZ. The strength values after the first hour of hold time prior to vacuum bag application were marginal and the flow test (acrylic plate with dry glass threads) exhibited only partial impregnation. The remaining panels, pressured at 2 hours and 3.5 hours, did not separate easily but were structurally unacceptable. It was obvious that pressure must be applied within 0.5 hour to achieve a satisfactory bond.

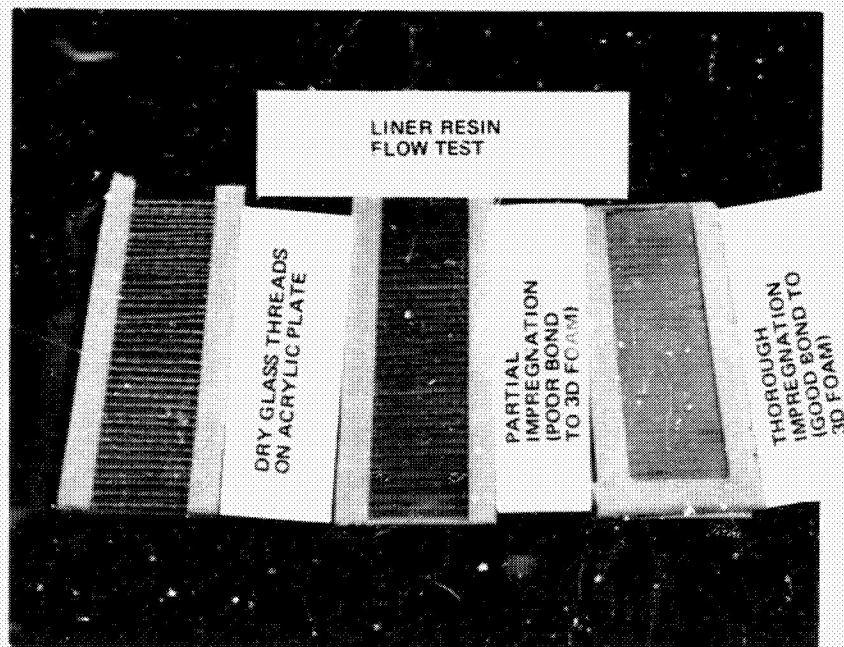
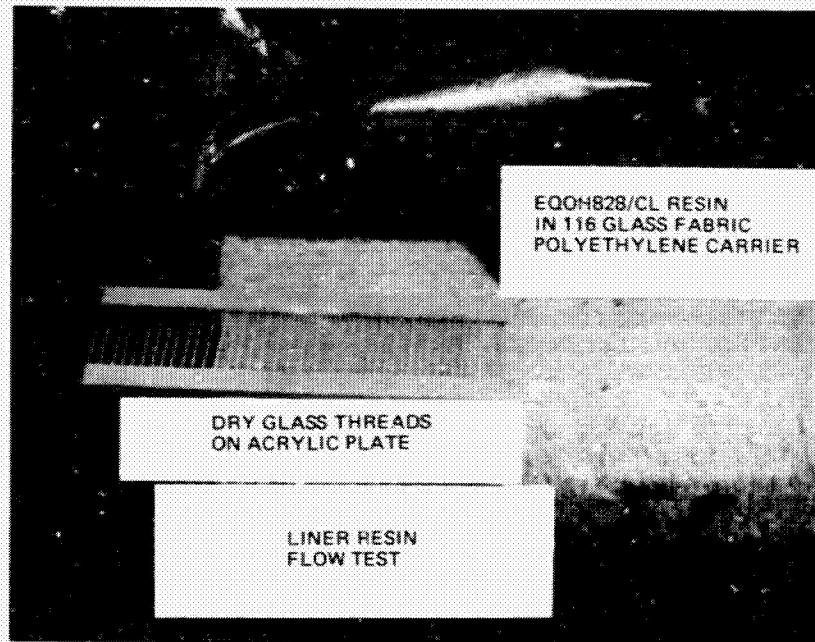


Figure 20. Liner Resin Flow Test

At a 43°C (110°F) holding temperature the liner bond strength was acceptable after 3.0 hours but dropped drastically at 4.0 hours. The remaining panels were discarded. The flow plate threads were completely impregnated on the corresponding 3.0 hour panel and exhibited partial or no impregnation on the longer hold time panels at 43°C (110°F).

At a 25°C (77°F) hold temperature the panels were pressured at 17, 21, 24, 26, and 30 hours. The strength values were comparable for panels representing 17, 21 and 24 hours. The 26 hour panel exhibited only spot bonding with the 3D foam and was discarded along with the 30 hour panel. The flow plate threads were completely impregnated on the corresponding 17 hour and 21 hour panels but a few dry threads could be detected on the flow plate representing the 24 hour panel. Dry threads were pronounced on the 26 and 30 hour plates.

At a 2°C (25°F) hold temperature the liner-3D foam panels were pressured at 48, 72, 93 and 120 hours. Strength values of panels up to a 93 hour hold time were normal and acceptable. Even the 120 hour panel exhibited a small degree of flow although this panel was discarded. The polymerization of Epon 828/CL was obviously arrested at 2°C (35°F) to a greater degree than observed with Lefkoweld 211A/LZ. It was not deemed necessary to conduct hold time tests between 93 and 120 hours because Lefkoweld 211A/LZ hold times are shorter and would regulate the manufacturing operation time.

Additional panels were tested using 13°C (55°F) holding temperature and validated the acceptable hold time at 13°C (55°F) to be 48 hours.

The readings taken on hardness specimens of Epon 828/CL at temperature above 18°C (65°F) were difficult to correlate with bond strength values because only a slight increase from Shore A of zero occurred at the initiation of resin gellation and Z fiber impregnation is reduced. The hardness readings of Epon 828/CL were useful at low temperature where resin having durometer reading, of over A40 did not soften at 52°C (125°F) sufficiently to completely impregnate the dry threads on the acrylic plate.

The thread impregnation test where fiber impregnation can be visually examined provided positive indication of bond strength, even though the visual results could not easily be converted to numbers.

The catalyzed working life curve showing the safe limits of operation along with hardness measurements and flow indication is presented in Figure 21.

2.3.4 Cold Hold Time Under Vacuum Bag Pressure (Delayed Start) to Warm Cure

The objective of this subtask was to determine the time limit between the time of application of vacuum bag pressure (cold tank wall) and the initiation of the warm cure cycle (under pressure).

Under normal manufacturing conditions, there would be a requirement to hold an area of positioned 3-D foam and liner under vacuum bag pressure until other segments of insulation could also be positioned and then conduct the warm cure cycle on a number of segments simultaneously. The previously conducted tests on this program have revealed that Lefkoweld 211A/LZ adhesive and Epon 828/CL liner will allow working life periods of ~72 hours, at 2°C (35°F) chamber temperature. Such a long working life period would allow at least half the entire tank surface to be covered with 3-D foam and liner then pressure applied by vacuum bag. Immediate initiation of the warm cure cycle would be expeditious at this point while preparing for the remaining half of the insulation operation. Thus the requirement for multiple bag installations, and delayed cure requirement while holding vacuum bag pressure would be relieved. However, contingency must be provided for malfunction of the heating equipment or other factors that could demand a holding period under vacuum pressure. Accordingly, strength tests were conducted on both liner and tank wall bond lines after holding at 25°C (77°F), 13°C (55°F), and 2°C (35°F) under vacuum bag pressure. To represent the most severe condition that could be expected during production bonding operations, the vacuum bag pressure was applied to these test panels at the end of the recommended catalyzed working life at each temperature and then held at that temperature for various time periods before initiating the warm cure cycle. All panels were then warm cured at 52°C (125°F) for 16 hours, still under vacuum bag pressure, then postcured at 149°C (300°F) without the vacuum bag in the standard manner. The test specimens for tensile bond

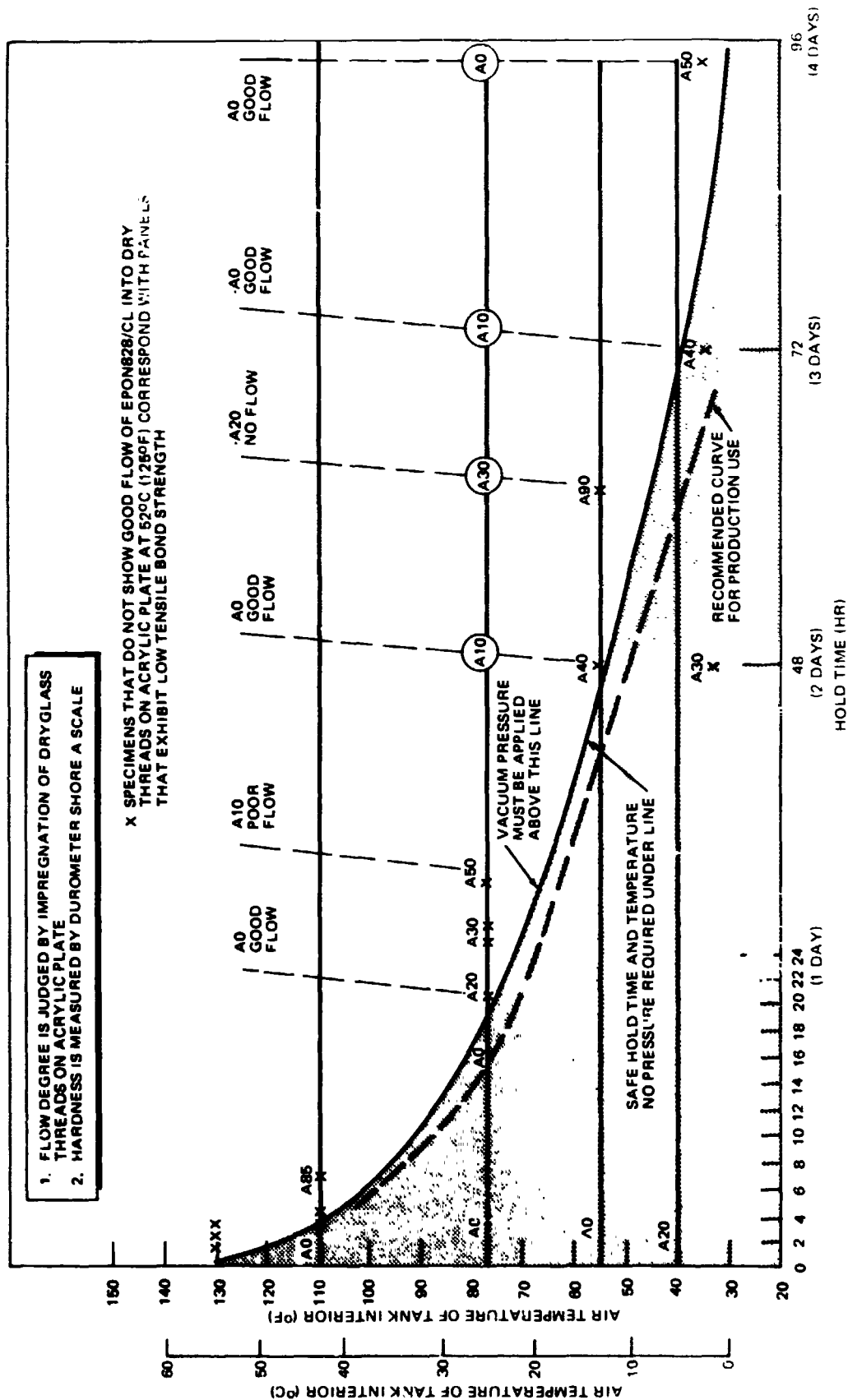


Figure 21. Catalyzed Working Life of EPON 828/CL Liner

strength determination at -196°C (-320°F) and at elevated temperature were prepared and tested in the manner previously described.

Over 30 panels were prepared for this series of tests, 10 panels at each of 3 temperatures; however, only a few of these panels were worthy of testing for tensile bond strength determination. These strength values are reported in Tables 9, 10, and 11. It quickly became apparent that the rate at which the panel was warmed to 52°C (125°F) after a prolonged hold period was more critical in achieving acceptable flow and bondability in the adhesive than whether vacuum bag pressure was applied or not applied at cold temperatures. The erratic spread in strength values of those panels actually tested, along with those panels not worthy of testing, suggested the warm cure cycle should be initiated at or before the time limits established for the catalyzed working life previously presented in Figures 19 and 21. Accordingly, it is recommended that no specific limits be established governing the time-temperature that can be allowed after the catalyzed working life period is exceeded and before initiating the warmup cycle to cure at 52°C (125°F) under vacuum bag pressure. In the event the warm cure cycle cannot be initiated within the limits prescribed for the catalyzed working life during production bonding operation, this condition must be considered as a discrepancy and judged by hardness determinations, flow examinations, and tensile bond strength of representative test panels. This analysis applies to both 211A/LZ adhesive and Epon 828/CL liner bond lines.

2.3.5 Warm Cure Temperature Limits

The objective of this subtask was to determine the minimum temperature and corresponding time cycle that will develop sufficient adhesive (211A/LZ) strength to hold the insulation composite in position against the tank wall and allow removal of vacuum bag pressure. The same situation exists for the Epon 828/CL impregnated glass fabric liner bonded to the Z fiber ends of 3-D foam. Both of these bond lines must hold firmly in position after the warm cure cycle and must endure personnel handling and postcure temperatures without the benefit of applied vacuum bag pressure.

During the warm cure cycle, personnel will be required inside the tank to monitor vacuum gages and correct leaks in the vacuum bag. For this reason,

Table 9
TANK WALL ADHESIVE HOLD UNDER
VACUUM BAG PRESSURE

Hold Prior to Vacuum Bag		Hold Under Vacuum Bag		Tensile Strength at -196° C (-320° F)	Temperature at Rupture Under Stress of 0.70 MN/m ² (100 psi)
Time (hr)	Temperature °C (°F)	Time (hr)	Temperature °C (°F)		
17	25 (77)	68	25 (77)		76 (168)
					153 (308)
					106 (222)
48	13 (55)	17	13 (55)	0.88 (127)	171 (340)
				1.59 (230)	174 (345)
				0.83 (120)	
				1.93 (280)	
48	13 (55)	61	13 (55)	1.45 (210)	171 (340)
				2.20 (318)	163 (325)
					169 (335)
72	2 (35)	144	2 (35)	1.38 (200)	168 (335)
				1.23 (178)	171 (340)
				0.83 (120)	176 (348)

the tank temperature should be as low as possible and the time required to reach the desired adhesive strength should be as short as possible.

The temperature conditions selected for examination were 25°C (77°F), 38°C (100°F), and 52°C (125°F). Higher temperatures were not considered acceptable for personnel working conditions. All test panels and hardness specimens were prepared using the standard mixing and coating procedures and were placed in the cold hold chambers to receive two separately conducted, no-contact, cold hold periods of 17 hours at 25°C (77°F) on one set and 72 hours at 2°C (25°F) on another set. The panels were then placed in vacuum bags and pressured at 0.068 (20) to 0.08 MN/m² (24 inches of mercury).

Table 10
LINER HOLD UNDER VACUUM BAG PRESSURE

Hold Prior to Vacuum Bag		Hold Under Vacuum Bag		Tensile Strength at -196° C (-320° F)	Temperature at Rupture Under Stress of 0.70 MN/m ² (100 psi)
Time (hr)	Temperature °C (° F)	Time (hr)	Temperature °C (° F)		
17	25 (77)	48	25 (77)		157 (315)
					157 (315)
					160 (320)
17	25 (77)	68	25 (77)		144 (292)
					156 (312)
					141 (286)
72	2 (35)	144	2 (35)	1.10 (160)	
				0.97 (141)	
				1.39 (202)	
				1.15 (167)	
				1.14 (166)	
				1.83 (266)	

2. .5.1 Results of Tests Conducted with Lefkowied 211A/LZ Adhesive Warm Cured at Various Temperatures

One group of 211A/LZ coated panels was allowed to set at 25°C (77°F), still under vacuum bag pressure, until the Shore durometer "A" scale registered over 90. This hardness point was reached after 8 hours in the case of the 25°C (77°F) cold hold panels, and within 5 hours in the case of the 2°C (35°F) cold hold panels. The vacuum bags were then removed and these panels were warmed slowly to 52°C (125°F) as the first step to a postcure at 149°C (300°F). The adhesive hardness immediately dropped to less than A50 (soft), and the panels separated at the edges.

This series was repeated using longer time periods under vacuum pressure in the attempt to develop sufficient polymerization at 25°C (77°F) to allow

Table 11
LINER STRENGTH AFTER CURE WITHOUT
VACUUM BAG^a

Hold Prior to Vacuum Bag		Hold Under Vacuum Bag		Temperature at Rupture Under Stress of 0.70 MN/m ² (100 psi)	
hr	°C (°F)	hr	°C (°F)	°C	(°F)
17	25 (77)	48	25 (77)	159	(318)
				<u>164</u>	<u>(328)</u>
				Average 162	(323)

^aAfter the hold under vacuum bag, the vacuum bag was removed and the specimens were given the standard cure and postcure cycle without a vacuum bag.

postcure at elevated temperature without having the vacuum bag in place. The 25°C (77°F) warm cure experiments were terminated after a 32-hour period under vacuum pressure proved to be unsuccessful in producing a panel worthy of strength evaluation.

The next group of 211A/LZ adhesive coated panels was placed in an oven at 43 ± 3°C (110°F ± 5°F) still under vacuum bag pressure. The time to reach hardness A90 was 5 hours in the case of the 25°C (77°F) cold hold panels and 4 hours in the case of the 2°C (35°F) cold hold panels. The vacuum bags were then removed and these panels were slowly warmed to 52°C (125°F) as the first step to post cure at 149°C (300°F). The adhesive hardness dropped to A70 and the panels separated at the edges. The 43°C (110°F) warm cure experiments were terminated after a 32-hour period under vacuum bag pressure proved to be unsuccessful in producing a panel worthy of strength evaluation.

The next group of 211A/LZ adhesive coated panels was placed in an oven at 52°C (125°F) still under vacuum bag pressure. The time to reach hardness A90 was 4 hours in the case of the 25°C (77°F) cold hold panels and 3 hours

in the case of the 2°C (35°F) cold hold panels. The vacuum bags were removed and these panels were slowly warmed to 60°C (140°F) as the first step to postcure at 149°C (300°F). The adhesive hardness dropped to A80 and although separation did not occur at this point a serious weakening of the bond line must have occurred because both of the panels ruptured during machining to prepare tensile bond strength specimens. Very slight depressions were visible at the Z-fiber contact points when the smooth surface of the adhesive was examined. The metal surface was clean and virtually free of adhesive particles.

Considerable data already existed on panels having the 52°C (125°F) warm cure under vacuum pressure for a 16-hour period. This was the standard cure cycle used for adhesive mixing evaluations. It was also known that a slow heating cycle over a period of 3 hours to reach 93°C (200°F) was required to maintain hardness over A100 at all times. From 93°C (200°F) to the post cure temperature of 149°C (300°F), the heating rate was not critical but was given a standard time of 2 hours. Further experiments to reduce these time periods would quickly reach a point of diminishing returns; therefore, the standard warm cure cycle of 16 hours at $52 \pm 3^\circ\text{C}$ ($125^\circ\text{F} \pm 5^\circ\text{F}$) is recommended for retention, with the additional requirement to maintain durometer hardness Shore A above 100 at all times after the release of vacuum pressure.

2.3.5.2 Epon 828/CL Liner

The identical series of tests described for 211A/LZ adhesive was also performed with the Epon 828/CL impregnated glass fabric bonded to 3-D foam. It was found that Epon 828/CL will polymerize slightly more rapidly than 211A/LZ although the difference is not significant. The CL hardener (Metaphenylene Diamine) used with Epon 828 epoxy resin reacts in a very similar manner as the LZ hardener used with Lefkowitz 211A epoxy adhesive.

The surface of the liner is visible at all times during the post cure cycle period and separations at the Z-fiber contact points are readily apparent as are surface waviness and other visual indications of poor bond to 3-D foam.

The warm cure experiments with Epon 828/CL liner performed at 25°C (77°F) and 43°C (110°F) under vacuum bag pressure were no more successful than with Lefkoweld 211A/LZ in achieving a quality bond to 3-D foam worthy of strength evaluation. Only after warm cure at 52°C (125°F) under vacuum bag for 16 hours could a measure of confidence be obtained that a postcure cycle at 149°C (300°F) would be successfully performed without vacuum bag pressure applied.

Accordingly, the same warm cure requirements recommended for Lefkoweld 211A/LZ are also recommended for the liner impregnated and bonded to 3-D foam using Epon 828/CL resin.

2.3.5.3 Heatup Rate to Warm Cure Temperature Under Vacuum Bag Pressure

The curves showing the time limit until vacuum pressure must be applied (catalyzed working life) to achieve a satisfactory bond were generated using a 52°C (125°F) initial cure cycle maintaining 0.068 MN/m² (20 in. of Hg) pressure until the Lefkoweld 211A/LZ, or Epon 828/CL liner, reached a hardness of at least Shore A100 measured at 49°C (125°F). The warm-up time to reach 49°C (125°F) is critical since this temperature is necessary to soften the adhesive after a long period of retarded polymerization at low temperature.

Tests were conducted using warmup cycles of 5.5°C (10°F) per hour and 11°C (20°F) per hour. It happens that nearly all test panels fabricated for other purposes were heated to 52°C (125°F) using a 2 hour warm-up period, starting at 2°C (35°F), or ~25°C (~45°F) per hour as measured on the aluminum panel surface enclosed in a vacuum bag. The oven reached 52°C (125°F) within 15 minutes but the test panel was wrapped with dry glass "bleeder" fabric and was resting on a plywood base, which retarded the heat transfer. The slower heating cycles of 5.5°C (10°F) and 11°C (20°F) proved to be too slow. The adhesive failed to soften and polymerization simply advanced without achieving flow. All six panels in this series separated easily at the termination of the final postcure at 149°C (300°F). Another two panels were then prepared to explore the effect of using a very rapid warmup period. The 2219-T87 aluminum plate, adjacent to the

adhesive-coated 3-D foam was exposed to the oven air preheated to 52°C (125°F) heating the aluminum plate to 49°C (120°F) within 5 minutes. These panels exhibited no higher tensile bond strength than achieved using the standard 2-hour heatup cycle. Accordingly, the 2-hour warmup period, or 25°C (45°F) per hour, was established as the minimum heatup rate. The control of heatup rate will also be governed by the additional requirement of achieving Shore A20 or less at 52°C (125°F) on the hardness specimens of Lefkowied 211A/LZ and complete flow of the Epon 828/CL liner to impregnate the dry glass threads on the acrylic plate described in Figure 20.

2.3.6 Vacuum Pressure Limits

The objective of this subtask was to establish the minimum vacuum pressure required to provide a structurally adequate bond and to determine criteria for rejection of the insulation in the event of loss of vacuum bag pressure.

2.3.6.1 Lefkowied 211A/LZ Tank Wall Bond

The standard procedure used in fabrication of test panels for other evaluations in this program used a vacuum pressure gage reading between 0.068 MN/m² (20) and 0.081 MN/m² (24 inches of mercury). Tests were conducted at 0.017, 0.034 and 0.051 MN/m² (5, 10, and 15 inches of mercury). The results of these strength tests are presented in Table 12 for the Lefkowied 211A/LZ tank wall bond strength. These tests were all conducted using flat panels and, as a result, very little pressure was needed to produce intimate contact between mating surfaces. Nevertheless, the strength values showed greater reliability is achieved at bonding pressure above 0.068 MN/m² (20 in. Hg) [although isolated strength values obtained on panels bonded at 0.034 MN/m² (10 in. Hg) are in the same range]. This would lend credence to the assumption that bonding pressure in excess of that required to assure contact between surfaces is necessary. Experience from Saturn S-IVB has shown that a minimum pressure of 0.068 MN/m² (20 in. Hg) is required to compensate for mismatches between tank wall contours and the 2.5 cm (1-inch) thick 3-D foam of 83 kg/m³ (5.2 PCF) density. The Shuttle 3-D foam having a density of 47 kg/m³ (2.7 PCF) is more flexible and will recontour more readily; however, the Shuttle tank wall mismatch may be greater and thus nullify the advantage of more flexibility in the 3D foam.

Table 12
 LOW-PRESSURE LIMIT OF TANK WALL ADHESIVE
 LOW LIMIT OF VACUUM BAG PRESSURE DURING BONDING
 OF 3-D FOAM TO TANK WALL

Gage Pressure		Tensile Strength at -196° C (-320° F)		Temper. are at Rupture Under Stress of 0.7 MN/m ² (100 psi)	
MN/m ²	(in. Hg)	MN/m ²	(psi)	° C	(° F)
0.017	(5)	0.75	(109)	152	(306)
		0.69	(100)	172	(342)
		0.70	(102)	167	(332)
0.034	(10)	2.54	(368)	161	(322)
		0.54	(78)	141	(286)
		1.44	(209)	180	(356)
0.051	(15)	0.69	(100)	184	(364)
		0.44	(64)	178	(352)
		1.73	(251)	179	(354)
0.068	(20) (nominal)	~1.72	(250)	~170	(340)

Establishment of 0.068 MN/m² (20 in. Hg) vacuum pressure gage indication as the minimum bonding pressure for Lefkoweld 211A/LZ adhesive between 3-D foam and tank wall will provide a reliable contact pressure and is practical to achieve using large vacuum bags sealed inside the tank.

2.3.6.2 Epon 828/CL-Glass Liner

The analysis applied to Lefkoweld 211A/LZ bonding appeared to hold also for bonding the Epon 828/CL resin impregnated 116 glass fabric liner to 3-D foam surface. The tensile bond strength values for liner-to-3-D foam are presented in Table 13 showing the effect of bonding pressures of 0.017, 0.034, 0.051 and 0.068 MN/m² (5, 10, 15 and 20 in. Hg). Again the strength values associated with panels fabricated under 0.068 to 0.081 MN/m² (20 to 24 in. Hg) pressure show greater reliability than was achieved with panels bonded using lower pressure although isolated strength values were of the same order of magnitude. Contour mismatch between the wet resin impregnated fabric and the 3-D foam obviously cannot occur with

Table 13
LOW-PRESSURE LIMIT OF LINER

Gage Pressure		Tensile Strength at -196° C (-320° F)		Temperature at Rupture Under Stress of 0.7 MN/m ² (100 psi)	
MN/m ²	(in. Hg)	MN/m ²	(psi)	° C	(° F)
0.017	(5)	0.84	(122)	44	(112)*
		0.79	(115)	66	(150)*
		0.97	(141)	-55	(130)*
0.034	(10)	1.03 ^a	(149)	157	(314)
		0.91 ^a	(132)	71	(160)*
		1.18 ^a	(171)	159	(318)
0.051	(15)	1.20 ^a	(174)	169	(336)
		1.29 ^a	(187)	109	(228)
		0.65	(94)	166	(331)
0.068	(20) (nominal)	~1.38	(200)	~166	(330)

Note. *Specimens ruptured from stress only. Not affected by temperature.

^aSpecimen failed primarily against tensile block or between tensile block and the liner.

these flat panels but does occur in tank insulation operations where the liner will bridge localized depressions in the 3-D foam surface. For this reason, the establishment of 0.068 MN/m² (20 in. Hg) vacuum pressure, gage indication, as the minimum bonding pressure for Epon 828/CL liner-to-3-D foam is recommended.

2.3.6.3 Intermittent Loss of Vacuum Pressure

During the warm cure cycle under vacuum bag pressure, the pressure may be inadvertently reduced as a result of bag leakage, hose collapse or disconnect, or pump malfunction. The causes are determined and corrected by personnel working within the tank; however, the brief period during which the vacuum pressure is reduced or entirely released can result in a structurally inadequate bond of the liner-to-3-D foam or between 3-D foam and tank wall.

The loss of vacuum pressure is critical only after the catalyzed working life is exceeded for the adhesive and liner. Therefore, all test panels used to determine the effect of pressure loss were first allowed to set in temperature controlled chambers at 25°C (77°F) for 17 hours and at 2°C (35°F) for 72 hours prior to closing together and applying vacuum bag pressure.

Each group of panels was subjected to two "leak" periods of 15-minute duration occurring at various temperatures during the warmup cycle to 49°C (120°F). Other panels were subjected to two "leak" periods after reaching the 52°C (125°F) warm cure temperature but within the first 2 hours while the adhesive was still soft.

A detailed description of each test condition is presented in Table 14.

The case history of these panels is also presented graphically in Figure 22.

The results derived from these tests presented in Tables 15 through 18 show that loss of vacuum pressure after the panel has reached 49°C (120°F) is extremely hazardous to both adhesive and liner bond lines and safeguards must be planned to avoid this discrepancy during production bonding operations. The complete loss of bond strength by loss of vacuum pressure at 49°C (120°F) during warmup from 2°C (35°F) would suggest the Lefkowitz 211A/LZ or Epon 828/CL is well advanced in polymerization at the end of the 72-hour cold hold at 2°C (35°F). On this basis, the 72-hour cold hold period at 2°C (35°F) should be reduced as suggested in Figures 19 and 21 to provide a greater margin of safety applied to production bonding operations.

Fortunately, the complete loss of vacuum pressure rarely occurs during production bonding operations. Vacuum bag leaks may occasionally result in reducing the bonding pressure below the required 0.068 MN/m² (20 in. Hg) limit, and Tables 12 and 13 indicate a tolerance to a low-pressure condition can be established along the following lines.

- A. Inadvertent reduction in vacuum bag pressure below 0.034 MN/m² (10 in. Hg) while the warm cure temperature is above 43°C (110°F) cannot be tolerated while the hardness is below A100. The time period during which the vacuum bag pressure is below 0.068 MN/m² (20 in. Hg) must not exceed 15 minutes.

Table 14
SPECIMEN CURE HISTORY
INTERMITTENT LOSS OF VACUUM PRESSURE TEST

No.	History
1	Hold specimens for 17 hours at 25°C (77°F) without vacuum bag. Then vacuum-bag and hold for 6 hours at 25°C. Turn off vacuum for 15 min. Reapply vacuum for 16 hours, then turn off vacuum for another 15 min. Reapply vacuum and cure 16 hours at 49°C (120°F). Remove the vacuum bag and postcure 32 hours at 149°C (300°F).
2	Hold specimens for 17 hours at 25°C (77°F) without vacuum bag. Then vacuum-bag and warm to 44°C (110°F). Turn off vacuum for 15 min. Reapply vacuum for 30 min, then turn off vacuum for another 15 min. Reapply vacuum and cure 16 hours at 49°C (120°F). Remove vacuum bag and postcure 32 hours at 149°C (300°F).
3	Hold specimens for 17 hours at 25°C (77°F) without vacuum bag. Then vacuum-bag and warm panel to 49°C (120°F). Turn off vacuum for 15 min. Reapply vacuum for 30 min, then turn off vacuum for another 15 min. Reapply vacuum and cure 16 hours at 49°C (120°F). Remove vacuum bag and postcure 32 hours at 149°C (300°F).
4	Hold specimens for 72 hours at 2°C (35°F) without vacuum bag. Then vacuum-bag and warm to 25°C (77°F). Turn off vacuum for 15 min. Reapply vacuum for 30 min, then turn off vacuum for 15 min. Reapply vacuum and cure for 16 hours at 49°C (120°F). Remove the vacuum bag and postcure 32 hours at 149°C (300°F).
5	Hold specimens for 72 hours at 2°C (35°F) without vacuum bag. Then vacuum-bag and warm to 44°C (110°F). Turn off vacuum for 15 min. Reapply vacuum for 30 min, then turn off vacuum for another 15 min. Reapply vacuum and cure 16 hours at 49°C (120°F). Remove vacuum bag and postcure 32 hours at 149°C (300°F).
6.	Hold specimens for 72 hours at 2°C (35°F) without vacuum bag. Then vacuum-bag and warm panel to 49°C (120°F). Turn off vacuum for 15 min. Reapply vacuum for 30 min, then turn off vacuum for another 15 min. Reapply vacuum and cure 16 hours at 49°C (120°F). Remove vacuum bag and postcure 32 hours at 149°C (300°F).

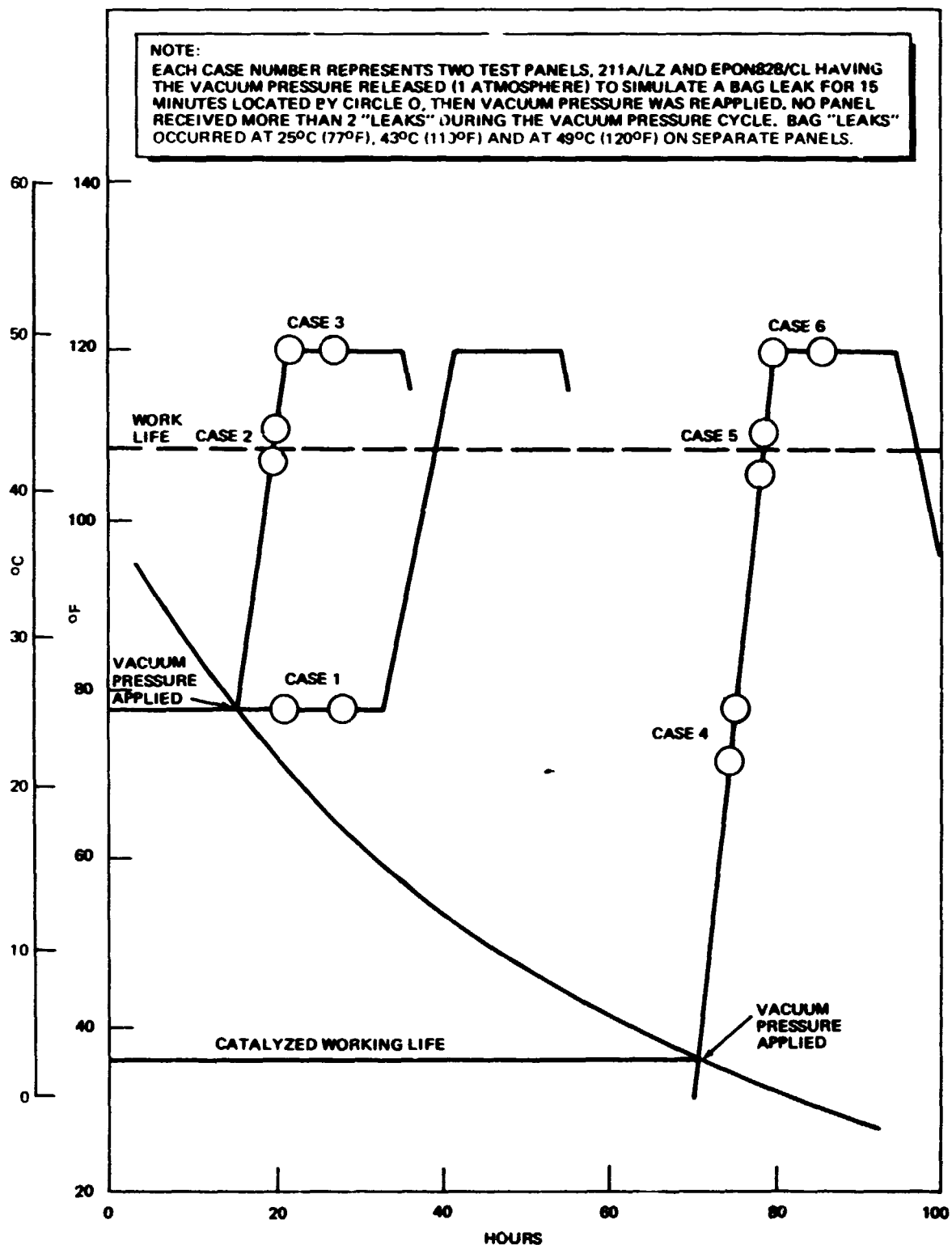


Figure 22. Intermittent Loss of Vacuum Pressure During Warm Cure Cycle

Table 15
TANK WALL ADHESIVE INTERMITTENT LOSS OF VACUUM
PRESSURE AFTER 25° C (77° F) HOLD

Specimen No. Cure History	Tensile Strength at -196° C (-320° F),		Temperature at Rupture Under Stress of 0.7 MN/m ² (100 psi)	
	MN/m ²	(psi)	°C	(°F)
1 Vacuum loss at 25° C (77° F) cold hold	1.16	(168)	138	(280)
	2.60	(376)	171	(340)
	1.10	(160)		
2 Vacuum loss at 43° C (110° F)	1.64	(238)	166	(330)
	1.30	(188)	174	(345)
	1.10	(228)	166	(330)
3 Vacuum loss at 49° C (120° F)	2.22	(322)	166	(330)
	1.69	(245)	176	(348)
	1.69	(245)		

Table 16
TANK WALL ADHESIVE INTERMITTENT LOSS OF VACUUM
PRESSURE AFTER 2° C (35° F) HOLD

Specimen No. Cure History	Tensile Strength at -196° C (-320° F),		Temperature at Rupture Under Stress of 0.7 MN/m ² (100 psi)	
	MN/m ²	(psi)	°C	(°F)
4 Vacuum loss at 25° C (77° F) during warmup from 2° C (35° F)	2.42	(350)	161	(322)
	0.52	(75)	168	(335)
	1.65	(240)	170	(338)
	0.86	(125)		
5 Vacuum loss at 43° C (110° F) during warmup from 2° C (35° F)	2.14	(310)	166	(330)
	2.40	(348)	176	(348)
	1.77	(256)	174	(345)
			171	(340)
Vacuum loss at 49° C (120° F) warmup from 2° C (35° F)	Panel separated after cure at 149° C (300° F)—No Bond			

Table 17
LINER INTERMITTENT LOSS OF VACUUM PRESSURE
AFTER 25°C (77°F) HOLD

Specimen No. Cure History	Tensile Strength at -196°C (-320°F)		Temperature at Rupture Under Stress of 0.7 MN/m ² (100 psi)	
	MN/m ² (psi)		°C	(°F)
1 Vacuum loss at 25°C (77°F) cold hold	0.97	(140)	110	(230)
	0.72	(105)	126	(260)
	0.86	(125)		
2 Vacuum loss at 43°C (110°F)	1.70	(246)	126	(260)
	1.76	(255)	152	(305)
	1.37	(198)		
3 Vacuum loss at 49°C (120°F)	1.69	(245)	154	(310)
	1.39	(202)	152	(305)
	1.42	(206)		

Table 18
LINER INTERMITTENT LOSS OF VACUUM PRESSURE
AFTER 2°C (35°F) HOLD

Specimen No. Cure History	Tensile Strength at -196°C (-320°F)		Temperature at Rupture Under Stress of 0.7 MN/m ² (100 psi)	
	MN/m ² (psi)		°C	(°F)
4 Vacuum loss at 25°C (77°F) during warmup from 2°C (35°F)	1.50	(218)	157	(315)
	1.53	(222)	160	(320)
	1.00	(145)	157	(315)
	0.92	(133)		
	1.52	(220)		
	1.12	(162)		
5 Vacuum loss at 43°C (110°F) during warmup from 2°C (35°F)	1.55	(225)	171	(340)
	1.63	(236)	157	(315)
	1.52	(220)	152	(305)
6 Vacuum loss at 49°C (120°F) warmup from 2°C (35°F)	Panel separated after 149°C (300°F) cure — No Bond			

- B. At warm cure temperatures below 43°C (110°F) the time period during which vacuum pressure is below 0.068 MN/m² (20 in. Hg) must not exceed 15 minutes.

2.3.7 Postcure Limits

The objective of this subtask was to determine the minimum time and temperature at elevated temperature necessary to establish a structurally adequate bond and to determine the allowable time span between warm cure and postcure.

If feasible, it is desirable to reduce the maximum postcure temperature below 149°C (300°F) to minimize metallurgical changes in the Shuttle tanks and to reduce the elevated temperature requirements of the heat-cure facilities. The use of lower postcure temperatures would also facilitate making insulation repairs.

Included in this subtask was a study to determine the effect of room temperature storage time between the warm cure and postcure operations. Tables 19 and 20 contain the tensile test results. The strength values indicate the final postcure cycle may be delayed for at least 70 days after completing the 52°C (125°F) warm cure cycle without affecting the bond strength.

Figures 23 and 24 summarize the results of a postcure time-temperature study. The data indicate that in order to achieve acceptable 177°C (350°F) strength for the tank wall adhesive, a postcure of at least 30 hours at 150°C (300°F) is required.

2.3.8 Other Related Work

During the period of reentry heating on the Shuttle LH₂ tank, the cold H₂ gas within the 3-D foam core may expand rapidly. The resultant pressure increase within the 3-D foam core could burst the composite insulation sandwich, unless the glass liner bonded to the 3-D foam is sufficiently porous to allow the expanding gas to escape easily. A study was made to measure the burst-pressure capability of the sandwich construction. In tensile bond strength testing of the liner-to-3-D foam using the MIL-STD-401 test method,

Table 19
TIME BETWEEN WARM CURE AND POSTCURE
OF TANK WALL ADHESIVE

Time Between Warm Cure and Postcure (days)	Tensile Strength at -196°C (-320°F) MN/m ² (psi)		Temperature at Rupture Under Stress of	
			0.7 MN/m ² °C	(100 psi), (°F)
21	1.16	(168)	171	(340)
	1.28	(185)	174	(345)
	1.93	(280)	171	(340)
56	2.37	(343)	172	(342)
	1.92	(278)	177	(350)
	2.76	(400)	154	(310)
77	2.28	(330)	179	(355)
	1.62	(235)	182	(360)
	1.77	(256)	179	(355)

Table 20
TIME BETWEEN WARM CURE AND POSTCURE
OF LINER

Time Between Warm Cure and Postcure (days)	Tensile Strength at -196°C (-320°F), MN/m ² (psi)		Temperature at Rupture Under Stress of	
			0.7 MN/m ² °C	(100 psi), (°F)
21	1.48	(215)	168	(335)
	1.52	(220)	149	(300)
	1.08	(156)	163	(325)
49	1.38	(200)	149	(300)
	1.05	(152)	134	(273)
	1.18	(171)	153	(308)
70	2.00	(290)	171	(340)
	1.12	(163)	179	(355)
	0.86	(125)	157	(315)

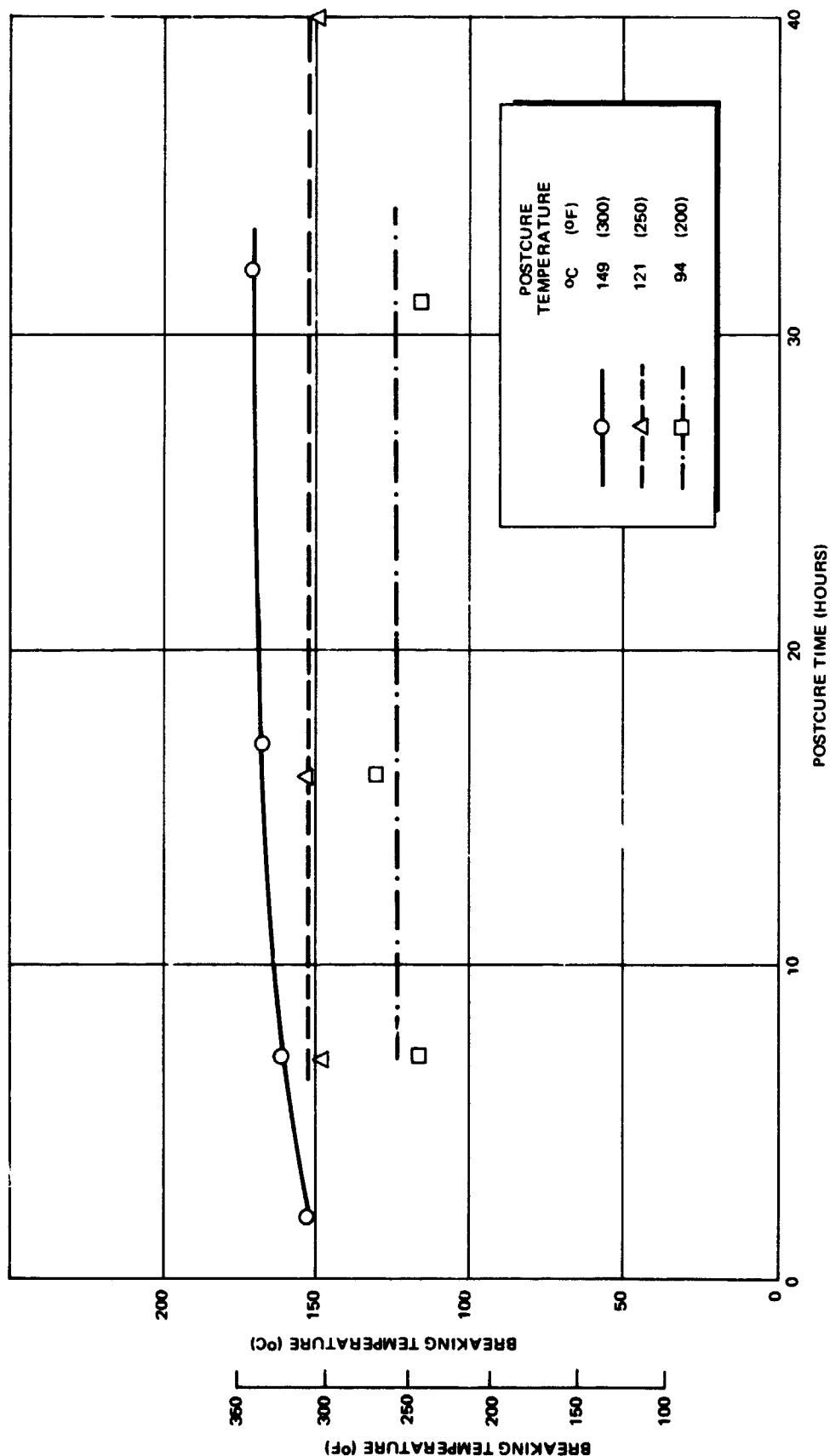


Figure 23. Tank-Wall Adhesive Lefkowitz 211A/LZ Postcure

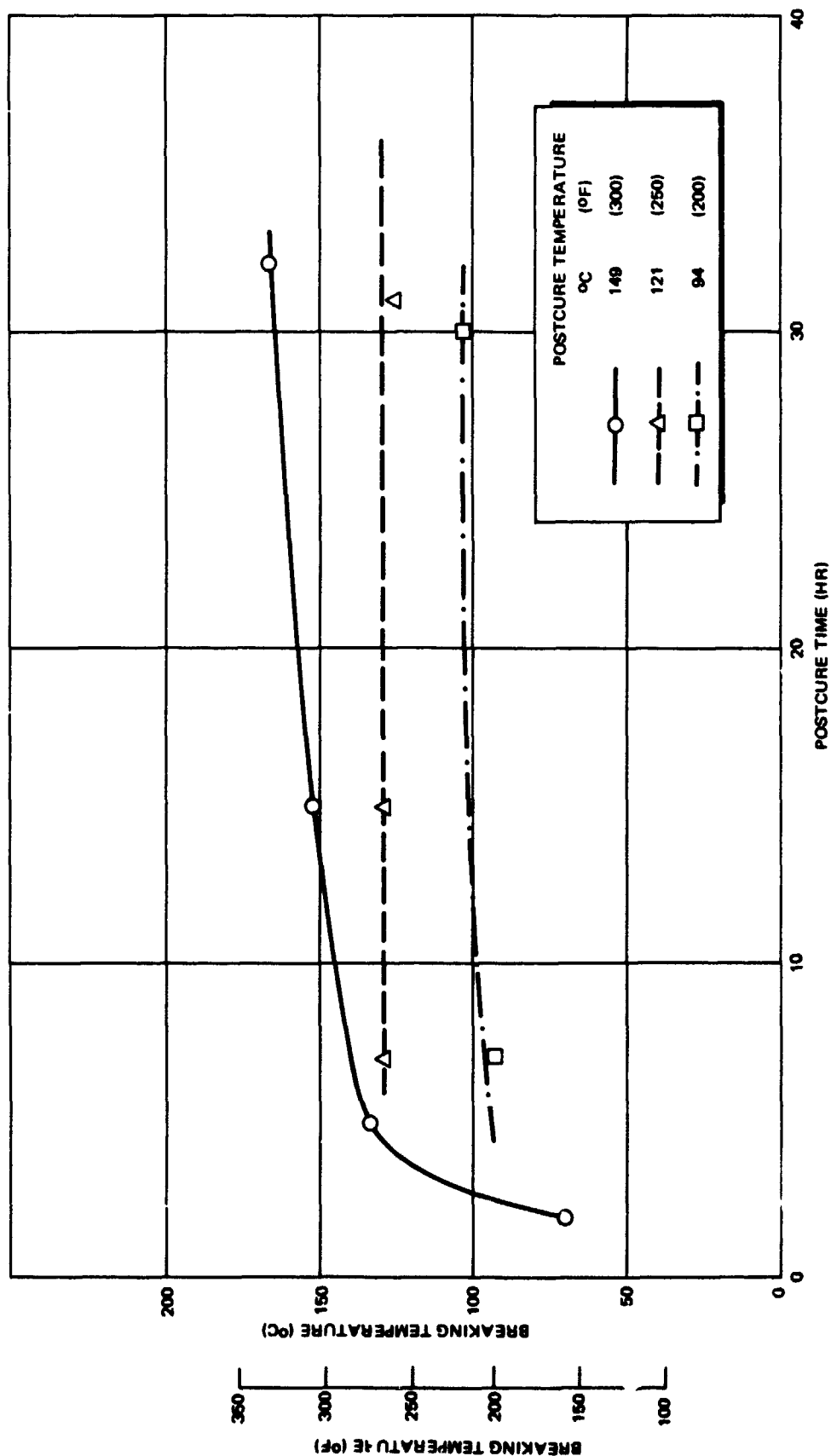


Figure 24. EPN 828/CL Liner Postcure

the liner is bonded perfectly flat against the pull block, a test which does not provide the peel effects that are induced by pneumatic pressure against the thin, glass liner.

Specimens were prepared (Figure 25) with an aluminum C-channel manifold bonded around the periphery of a square 3-D foam composite. The aluminum C-channel was provided with fittings to allow air to be introduced at one edge and the pressure to be monitored at the opposite edge, as shown in Figure 26. The air was introduced into the sandwich at 25°C (77°F). The air streams through the liner pores were audible at 0.035 MN/m² (5 psi) and readily discernible by finger touch. At 0.41 MN/m² (60 psi), the liner bond to the 3-D foam ruptured. Duration of test was approximately 30 sec.

A review of the initial test specimen size — 10.9 by 10.9 cm (4.3 by 4.3 in.) inside the manifold flanges — indicated that the test area of 119 cm² (18.49 in.²) may have been too small to represent the burst pressure capability of tank insulation in which the likelihood of bond defects is greater.

Accordingly, a second series of 30.5 by 30.5 cm (12 by 12 in.) panels was fabricated having a 684 cm² (106 in.²) test area inside the manifold flanges. One rub coat was used to seal the liner in accordance with standard procedure. Air pressure was introduced into the panels in the same manner as with the smaller test panels. With full open valve, both gages registered only 0.21 MN/m² (30 psi). No rupture was observed during these two tests.

In summary, these liner pressure tests indicate that liner-to-3D foam bond strength should be sufficiently high and liner porosity sufficiently high to prevent liner bond rupture as a result of rapid outgassing during reentry.

2.4 VIBRATION AND ACOUSTIC ANALYSIS

The objective of this task was to provide an analytical assessment of the effect of the Space Shuttle vibration and acoustic environment on the insulation system and to define a test plan for vibration and acoustic performance verification.

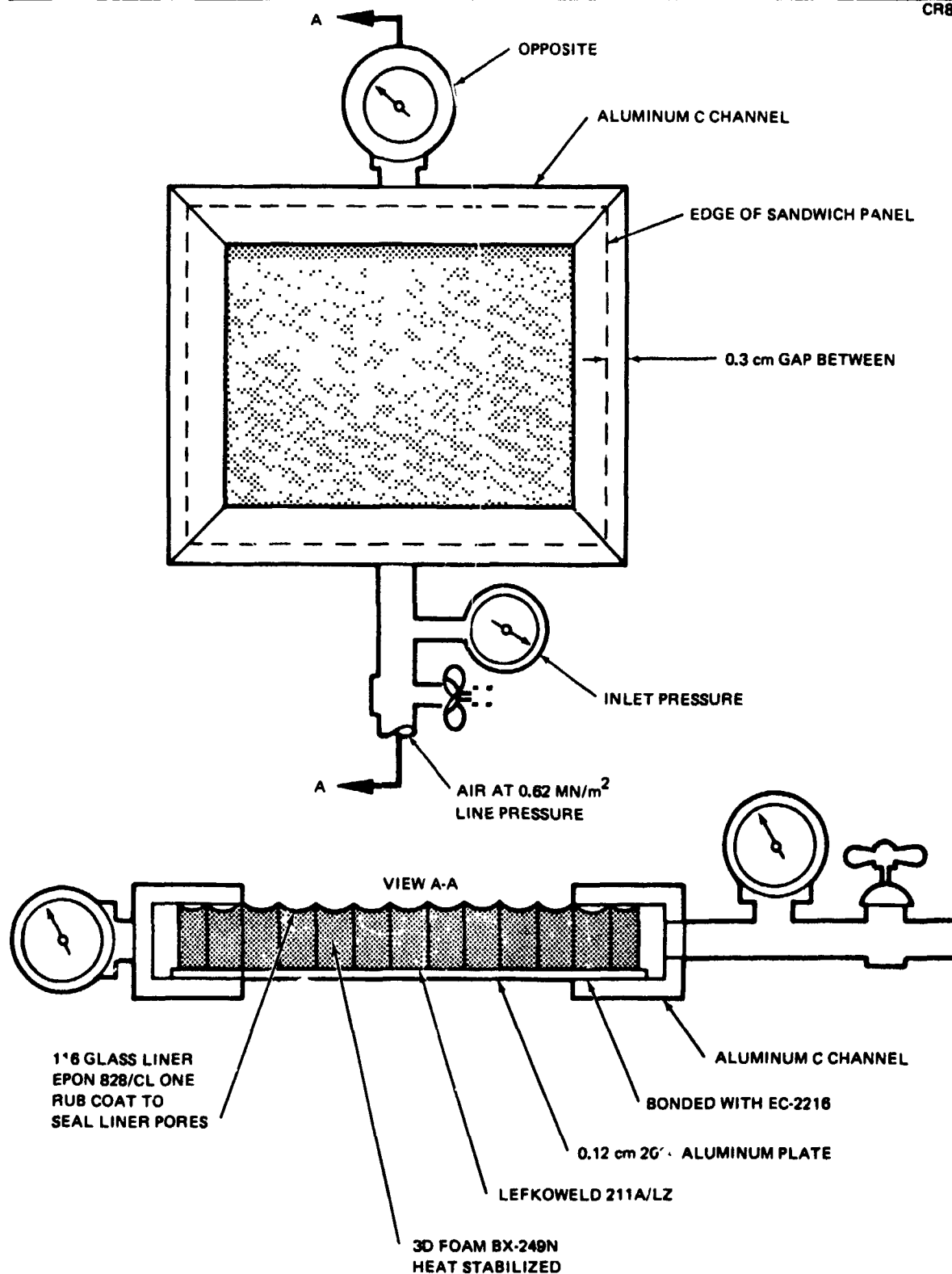


Figure 25. Liner Burst Test Setup

AT PLUS LINE BEARING
CAPTIVE LINE BEARING
AT 100 PSI BORE
72 1/2"

CHAMBER WEIGHT 100 LBS
1/2" DIA. 100 PSI BORE
1/2" DIA. 100 PSI BORE
1/2" DIA. 100 PSI BORE
1/2" DIA. 100 PSI BORE

71

The fatigue strength of internal 3-D foam insulation has been demonstrated to be adequate to withstand the combined vibration and low temperature environments for use in the LH_2 tank of the Saturn S-IVB stage. However, insufficient data exist to confirm its capability to sustain the long life requirements of a reusable Shuttle Booster or Orbiter vehicle. It is anticipated that a test program using beam and panel specimens will be required to investigate the long life fatigue strength of the insulation system. The effect of both low and high temperature on the fatigue life will also require investigation.

In order to define the most meaningful fatigue test, an analytical study was made to determine the effects of acoustically induced vibration of the insulation system. This study used existing dynamic analysis techniques which were developed for use on the S-IVB and Skylab Programs and verified by data on full-scale insulated tanks. These techniques were adapted to a heat sink configuration of the Shuttle booster. The stiffness and damping characteristics of the insulation were estimated based on experience with insulation on the programs mentioned above, and consideration of the form characteristics previously reported.⁴

The study consisted of the following:

- A. Definition of the expected acoustic and temperature environments.
- B. Analysis of the structural configuration to establish the cylinder response.
- C. Determination of the size of the fatigue test specimens and fatigue test conditions using the information from the analyses.

These steps are discussed in more detail on the following pages.

2.4.1 Environmental Definition

It is expected that three phases in the flight will cause significant acoustic levels. These are:

- A. Liftoff.
- B. Boost.
- C. Reentry.

In order to establish test conditions it is required that the expected external acoustic levels with the associated temperature ranges be determined. These combinations and other input data are tentatively identified in Table 21 for the tank configuration shown in Figure 27.⁵

For this structural analysis the booster liquid hydrogen tank was modeled as a 360 degree of freedom system. Each degree of freedom represented the deflection at the center of a rectangular panel of tank wall. Ten panels per axial row and thirty six total rows comprised the total of 360 panels. The dimensions of each panel were approximately 0.81m (32 inches) wide by 2.5m (100 inches) long.

Resonant frequencies of a cylinder accounting for internal liquid and pressure were determined by the method obtained from Reference 6. The cylinder at liftoff was dynamically modeled as being filled with liquid hydrogen at an internal pressure of 0.21 MN/m^2 (30 psi). The reentry condition consisted of an empty tank at 0.14 MN/m^2 (20 psi) pressure. At maximum dynamic pressure, the booster tank is approximately half filled with propellant.

Table 21
ENVIRONMENTAL DEFINITION FACTORS

Flight Condition	Insulation Temp. °C (°F)	Pressure Differential, MN/m^2	Liquid Level, m (in.)	Acoustic Environment, dB (O.A.)*	Effective Tank Thickness, cm (in.)
Liftoff	-253 (-423)	0.105 (15.3)	3.2 (820)	164.5	1.14 (0.4495)
Ma - Q	-253 (-423)	0.189 (27.4)	1.6 (410)	155.0	1.14 (0.4495)
Reentry	159 (310)	0.14 (20) (est)	0 (0)	154.5	0.943 (0.371)

*O.A. = Overall

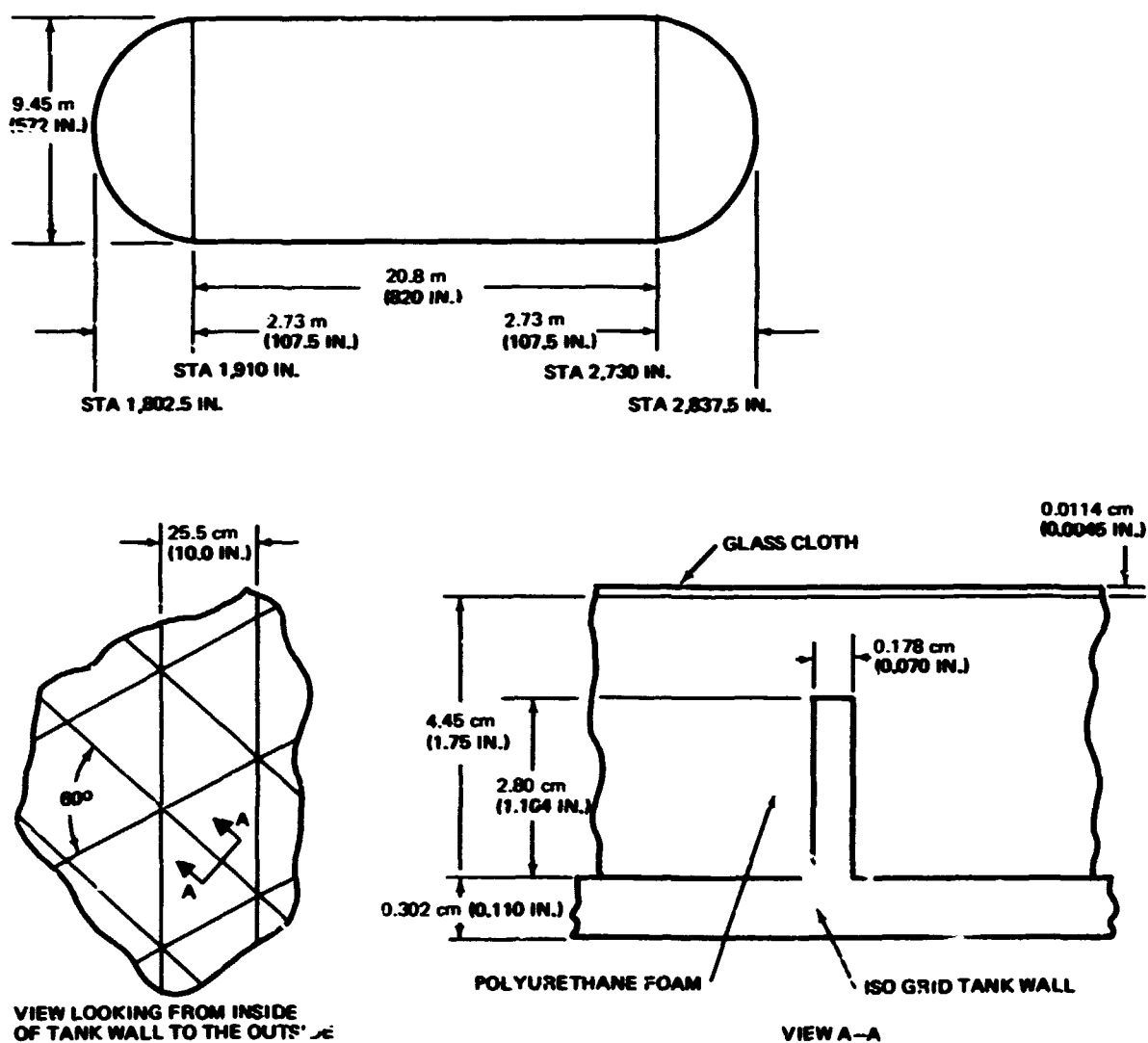


Figure 27. Configuration of Shuttle Booster Liquid Hydrogen Tank

Experimental data from Reference 6 indicate that a valid approximation is to consider each half of the tank separately. The empty portion of this tank was modeled for this analysis as it was expected to have higher stresses than the liquid filled portion. The resonant frequencies for liftoff, maximum dynamic pressure and reentry are shown in Figures 28 through 30.

The external acoustic environment on the booster hydrogen tank is shown in Figure 31. Figure 32 presents a curve of dynamic amplification factor versus resonant frequency which was assumed for this analysis. These dynamic amplification factors were derived from a comparison between analysis and flight data on the S-IVB liquid hydrogen tank.

The acceleration response of the tank wall for the first stage flight conditions was determined by solving the equation for $[R_s]$ on page 38 of Reference 6. The solution of $[R_s]$ gives the acceleration power spectral density (PSD) for each of the 360 degrees of freedom. The acceleration PSD's in (gravity units)²/Hertz for liftoff, maximum Q and reentry are presented in Figures 33, 34, and 35, respectively. The panel for degree of freedom (DOF) No. 1 lies along the forward edge of the cylinder. The symmetry of the tank means that all panels along the forward and rear edge have the same acceleration response as DOF No. 1. The panel for DOF No. 6 lies near the center of the tank wall in the first axial row of panels. Similarly, the symmetry condition means that the random vibration acceleration response for DOF No. 6 is the same for all 36 panels lying near the center of the tank.

Reference 6 also provides the procedure to determine a matrix of stresses in each panel for a unit deflection of each vibration mode. These stresses include both normal and shearing stresses. This matrix was designated σ_{unit} . The relationship between the random stress response and the acceleration PSD response, R_s is given by:

$$[\sigma_{\text{PSD}}] = \begin{bmatrix} 2 \\ \sigma_{\text{unit}} \end{bmatrix} \times [R_s] \times \begin{bmatrix} \frac{1}{4} \\ W_f \end{bmatrix}$$

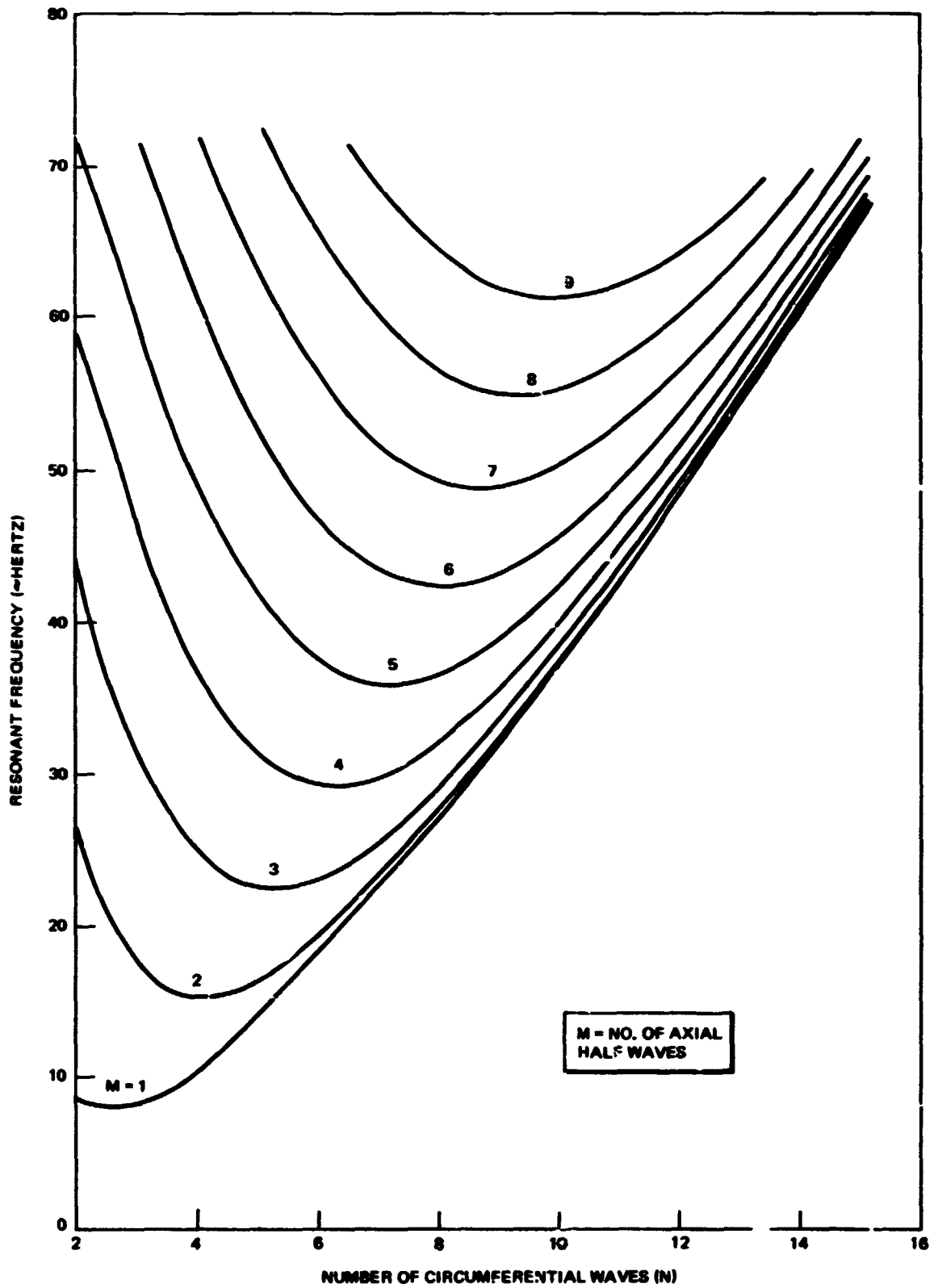


Figure 28. Resonant Frequencies of LH₂ Tank During Liftoff

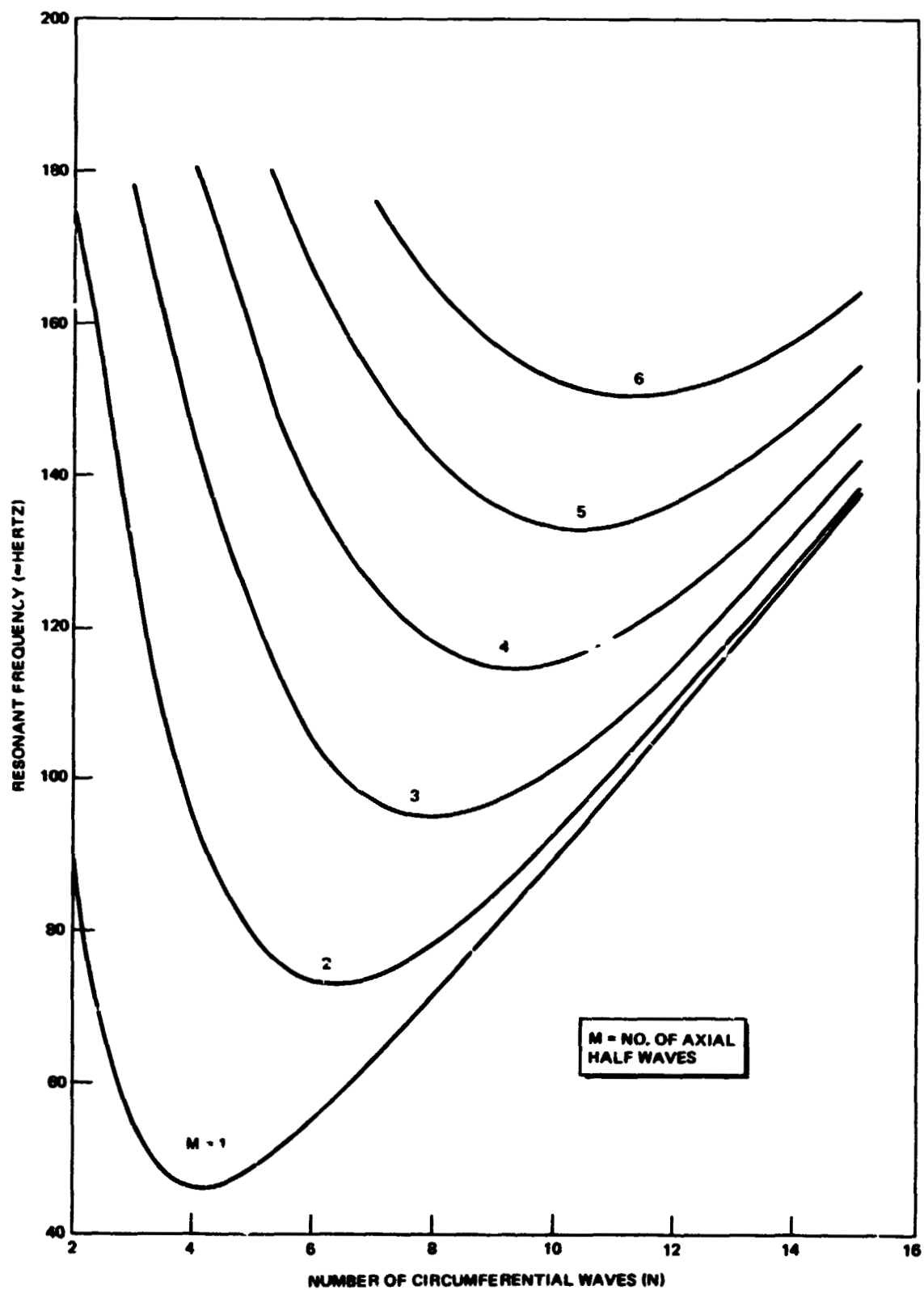


Figure 29. Resonant Frequencies of Upper Half of LH₂ Tank During Maximum Dynamic Pressure

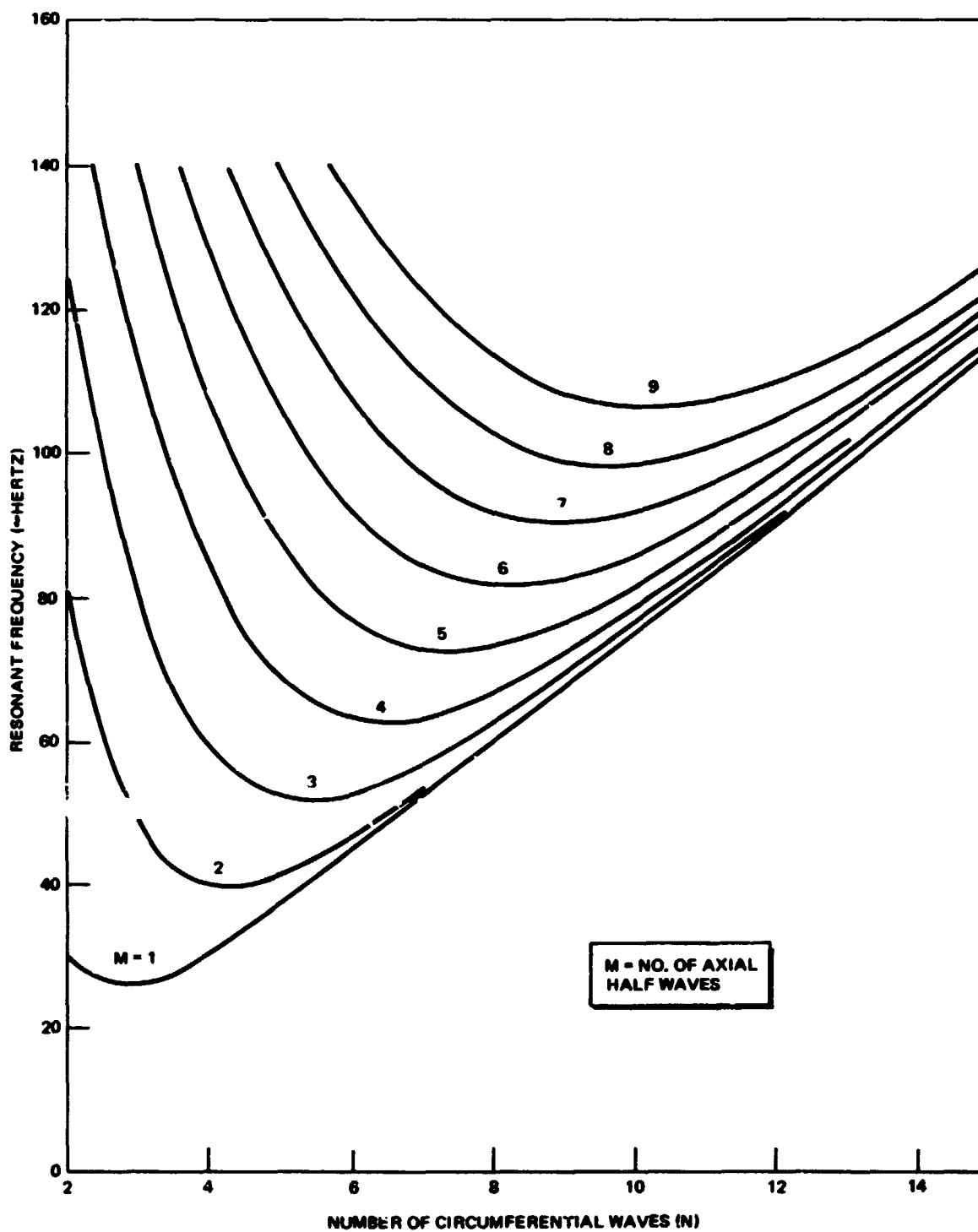


Figure 30. Resonant Frequencies at Reentry

ALICE 0375 PHASE B SPACE SHUTTLE, FINAL REPORT

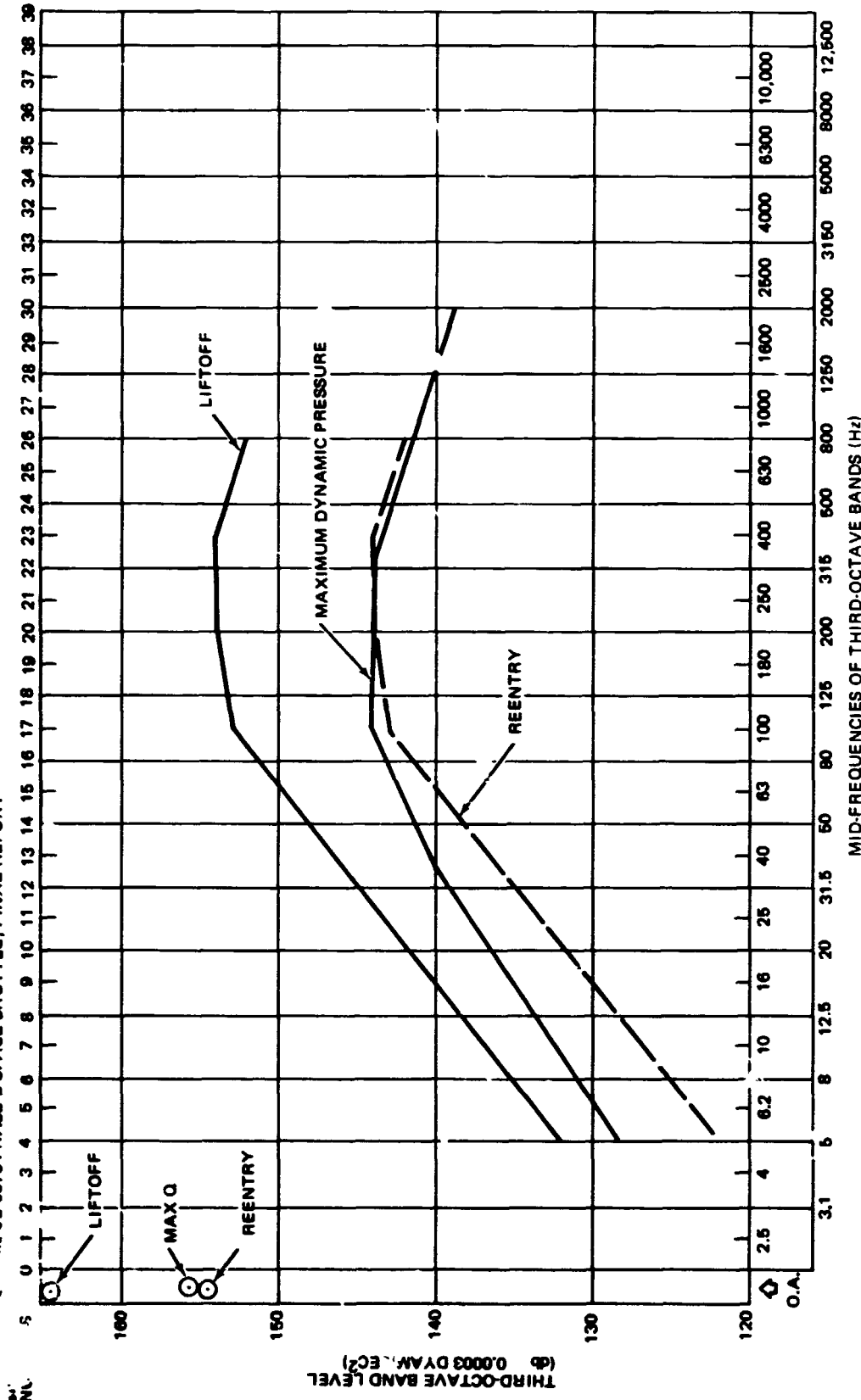


Figure 31. Predicted Acoustic Sound Pressure Levels for Shuttle Booster Hydrogen Tank

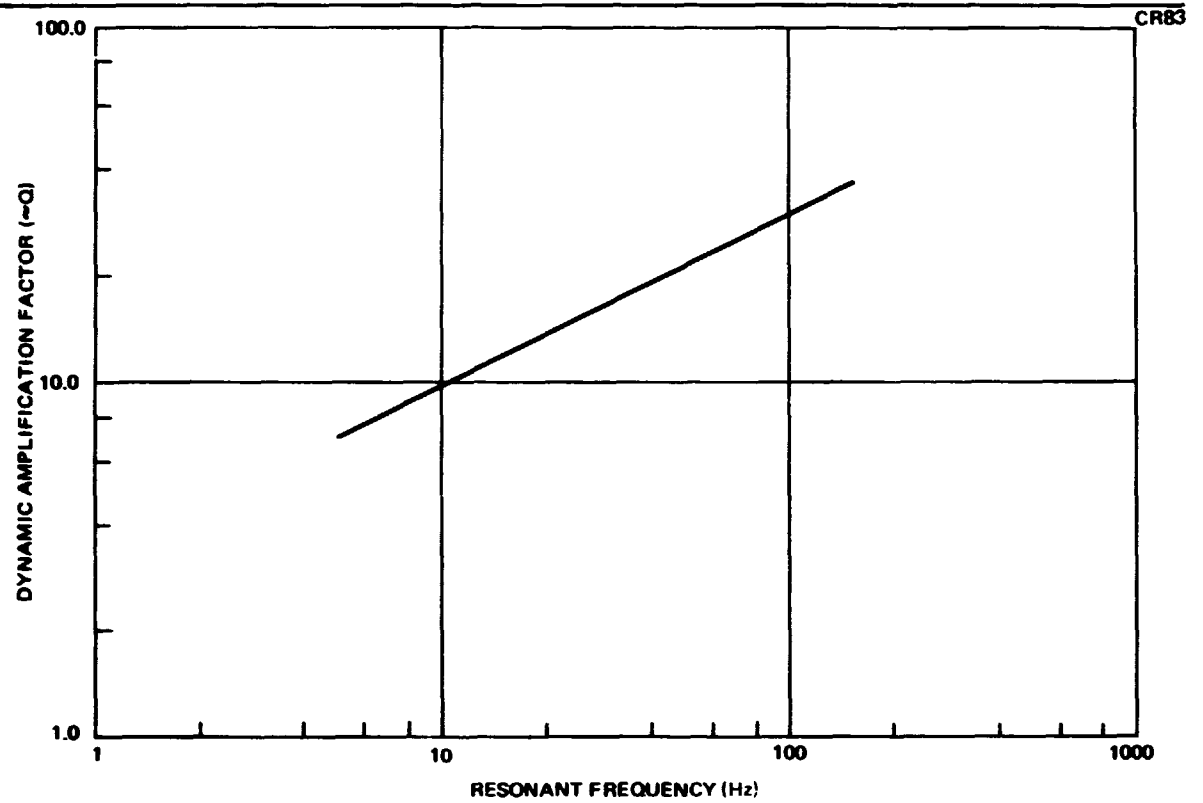


Figure 32. Dynamic Amplification Factor Versus Resonant Frequency of Booster LH_2 Tank Used in Analysis

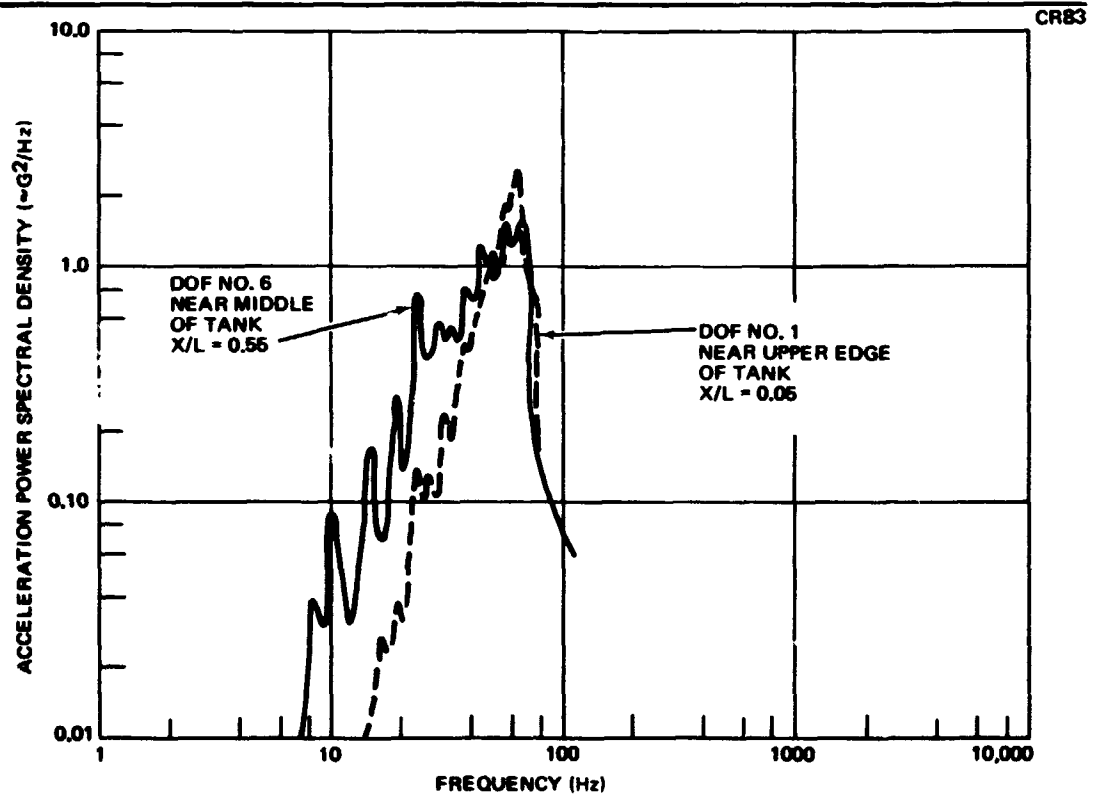


Figure 33. Acceleration Response at the Center and Edge of Shuttle Booster Hydrogen Tank at Liftoff

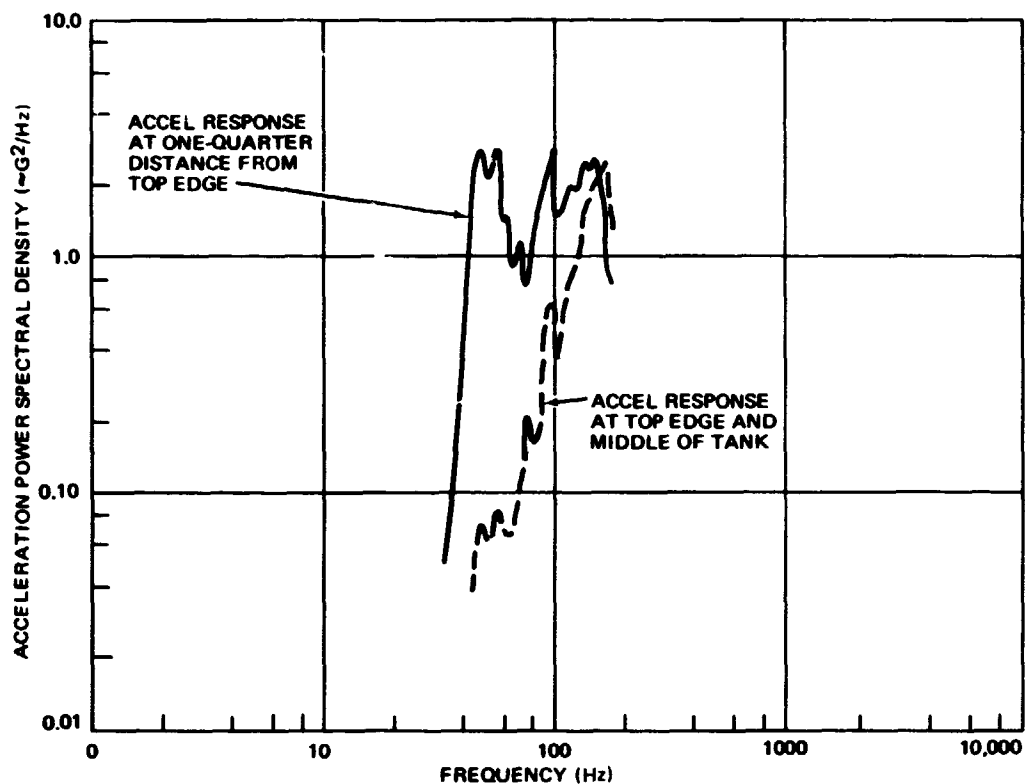


Figure 34. Maximum Acceleration PSD's on Tank Wall During Maximum Dynamic Pressure

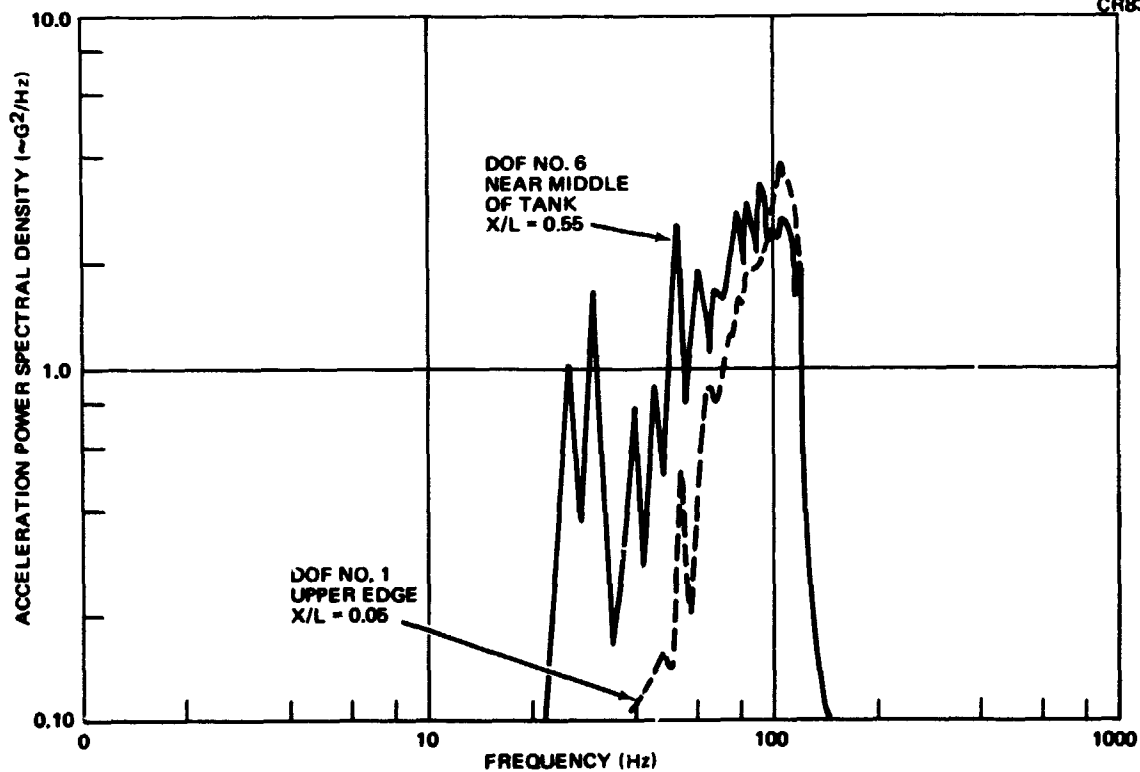


Figure 35. Acceleration Response at Center and Edge of Tank for Reentry Condition

$[\sigma_{\text{PSD}}]$ = stress PSD at each frequency, W_f

$[W_f]$ = diagonal matrix of frequencies at which stress PSD's are to be determined (radians/sec)

Figures 36 and 37 give a curve of stress PSD's during liftoff and maximum dynamic pressure for normal stresses in the x and y directions. The stress PSD's shown in these figures are the maximum values for the entire tank and occur at the center of the tank. Stress PSD's for the reentry condition were also determined but have not been shown. This curve is similar but of lower value than the liftoff stresses.

Integrating the stress PSD's with respect to frequency gives the root mean square (RMS) values of the dynamic stress. MDAC design policy is to consider an equivalent peak value of stress to be three times the RMS value. An equivalent fatigue stress is taken as twice the RMS value. This equivalent constant level fatigue stress produces the same fatigue damage per unit of time as the RMS random vibration stress. Equivalent limit and fatigue stresses in the tank wall and insulation are presented in Table 22 for liftoff, maximum dynamic pressure and reentry.

The predicted stresses in the composite insulation due to the dynamic environment would be used in a fatigue test program to determine the structural integrity of this insulation for shuttle applications.

2.4.2 Fatigue Test Definition

Using the information from the studies described above, a fatigue test plan was developed. Coupon tests using bending stresses equal to combined tensile and bending stress from the analysis were selected to verify the structural integrity of the advanced insulation. Fatigue tests combining bending and tensile stresses in the proper relationship would prove costlier and more difficult to control. Tests using tensile stress only would not simulate the bending loading conditions.

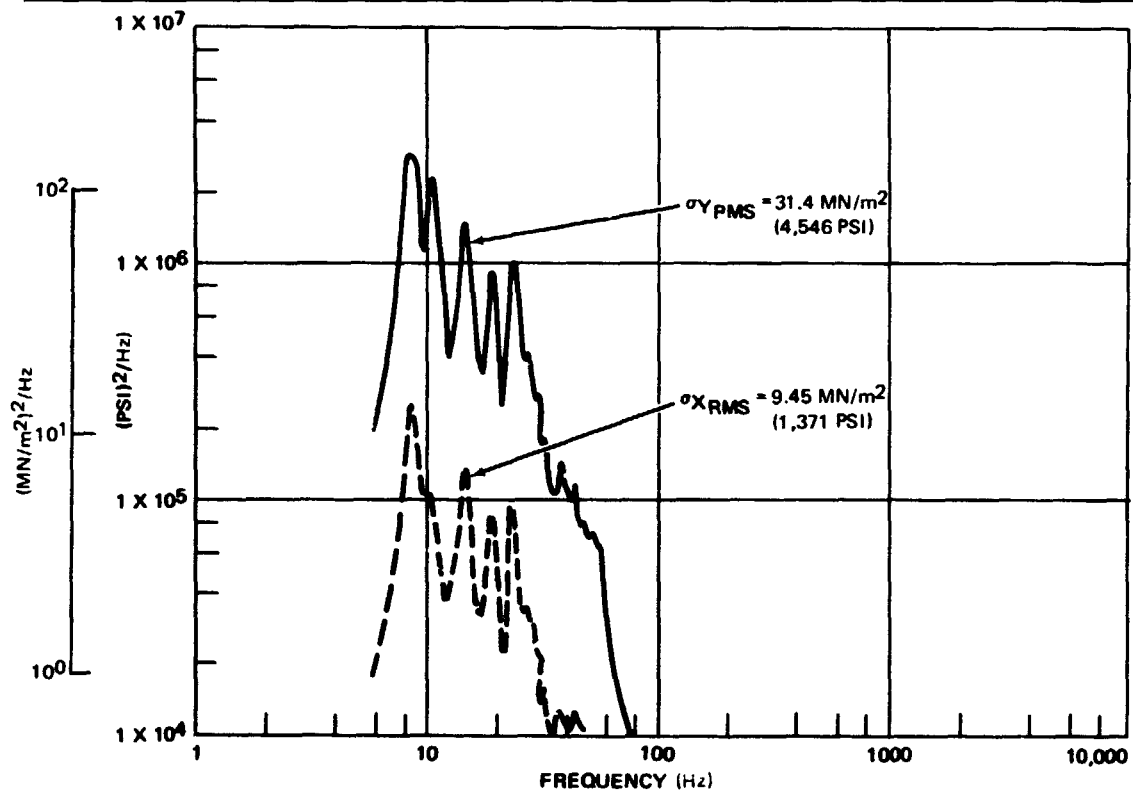


Figure 36. Maximum Stress PSD'S in Tank Wall During Liftoff

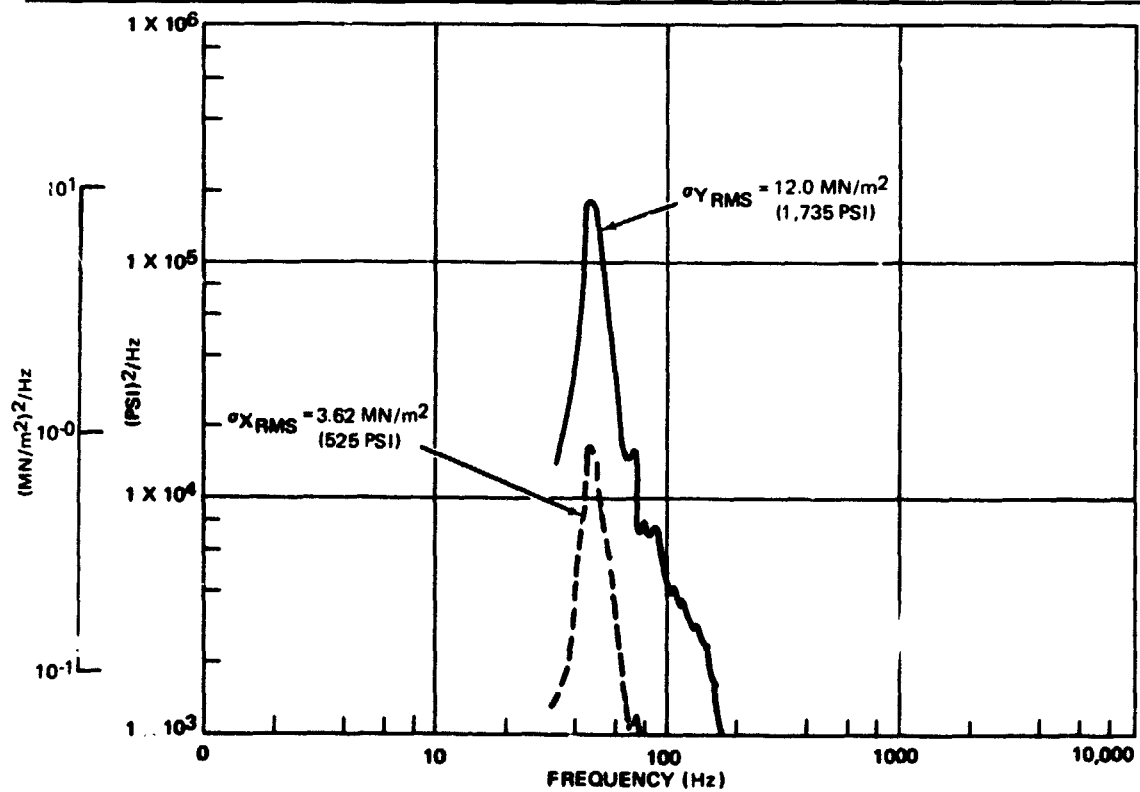


Figure 37. Maximum Stress PSD's in Tank Wall During Maximum Dynamic Pressure

EQUIVALENT LIMIT AND FATIGUE DYNAMIC STRESSES IN THE

	Equivalent Limit Stress, MN/m ² (psi)				
	x		y		xy
Liftoff					
Glass cloth liner, $Z = Z_2$	3.45	(522)	12.4	(1,801)	1.49
Polyurethane foam, $Z = Z_1$	0.0202	(2.92)	0.0676	(9.8)	0
Tank wall, $Z = Z_4$	28.4	(4,114)	94.0	(13,638)	0.555
Max Q					
Glass cloth liner	1.45	(210)	10.42	(1,513)	
Polyurethane foam	0.0077	(1.12)	0.026	(3.72)	0
Tank wall	10.86	(1,575)	35.9	(5,204)	0.331
Reentry					
Glass cloth liner	1.25	(182)	4.24	(615)	0.421
Polyurethane foam	0.0041	(0.60)	0.014	(2.0)	0
Tank wall	12.5	(1,814)	42.7	(6,196)	2.50

Number of Fatigue Cycles

Liftoff

= No. of Cycles

= 8 Cycles/Sec x 15 Secs/Miss

= 12,000 Cycles

Maximum Dynamic Pressure

= 47.0 Cycles/Sec x 30 Secs/Min

= 141,000

Reentry

= 26 Cycles/Sec x 60 Secs/Min

= 156,000 Cycles

Young's Modulus of Elasticity Used in A

(1) 2219-T86 Aluminum 68.9×10^3 psi(2) Poly. Foam 50.4 MN/m²(3) Glass Cloth Liner 12.4×10^3 psi

Table 22
STRESSES IN THE SHUTTLE BOOSTER HYDROGEN TANK CYLINDER

MN/m ² (psi)		Equivalent Fatigue Stress, MN/m ² (psi)			
	xy	x	y	xy	
801)	1.49 (216)	2.41 (349)	8.28 (1,201)	0.993 (144.0)	
(9.8)	0	0.0134 (1.95)	0.0448 (6.5)	0	
638)	0.555 (80.5)	18.9 (2,743)	6.27 (9,092)	0.371 (53.7)	
513)		0.775 (112.0)	6.95 (1,008)	0.517 (75.0)	
(3.72)	0	0.0052 (0.75)	0.017 (2.47)	0	
204)	0.331 (48.0)	7.25 (1,051)	26.3 (3,817)	0.221 (32.0)	
615)	0.421 (61.0)	0.835 (121.0)	2.83 (410.0)	0.276 (40.0)	
(2.0)	0	0.0028 (0.4)	0.0097 (1.4)	0	
196)	2.50 (362.0)	8.33 (1,209)	28.5 (4,131)	1.66 (241.0)	

cles

les

Sec x 15 Secs/Mission x 100 Missions

cles

c Pressure

Sec/Sec x 30 Secs/Mission x 100 Missions

Sec x 60 Secs/Mission x 100 Missions

cles

asticity Used in Analysis

	Liftoff		Reentry	
um	68.9 x 10 ³ MN/m ²	(10.0 x 10 ⁶ psi)	65.5 x 10 ³ MN/m ²	(9.5 x 10 ⁶ psi)
	50.4 MN/m ²	(7,300 psi)	20.7 MN/m ²	(3,000 psi)
	12.4 x 10 ³ MN/m ²	(1.8 x 10 ⁶ psi)	6.89 x 10 ³ MN/m ²	(1.0 x 10 ⁶ psi)

The highest dynamic stresses in the cylinder wall and insulation resulting from the shuttle acoustic environment are due to the low frequency vibration modes of the tank cylinder. In these vibration modes the dynamic stresses are composed of 75 percent tensile membrane and 25 percent bending stress.

The general approach selected would be to determine fatigue allowables at two extreme temperatures using beam coupon tests. Tentative test temperatures of -253°C (-423°F) and 150°C (300°F) were selected. A cryogenic test temperature of -196°C (-320°F) might be substituted for -253°C in order to reduce costs.

A total of 8 beam coupons would be tested to failure to establish an approximate fatigue curve for both the hot and cold temperature environments. Three specimens would be tested at each temperature. Figure 38 shows the configuration of the beam bending coupon specimen and test installation. The initial stress levels and number of fatigue cycles to be applied to the first specimen for each temperature condition are presented in Table 23. Table 24 shows the expected dynamic forces to be applied to the first beam specimens to obtain the fatigue stresses listed in Table 23. Strain gages would monitor the actual strain in the glass cloth liner and tank wall and the applied dynamic load would be adjusted to maintain the fatigue stress in the glass cloth liner to the values given in Table 23.

The first specimen for each temperature condition would be tested at the stress levels in Table 23 until failure; or until the number of cycles from Table 23 are accumulated. In the absence of fatigue failure after the first set of cycles, the stress levels would be raised by 50 percent and testing continued until failure or an additional number of cycles equal to the first set have been applied.

Testing would continue by repetitively raising the stress level 50 percent and applying the number of cycles listed in Table 23 until failure occurs. The number of fatigue cycles listed in Table 23 is equal to the amount incurred during each flight condition for a total of 300 flights. This duration

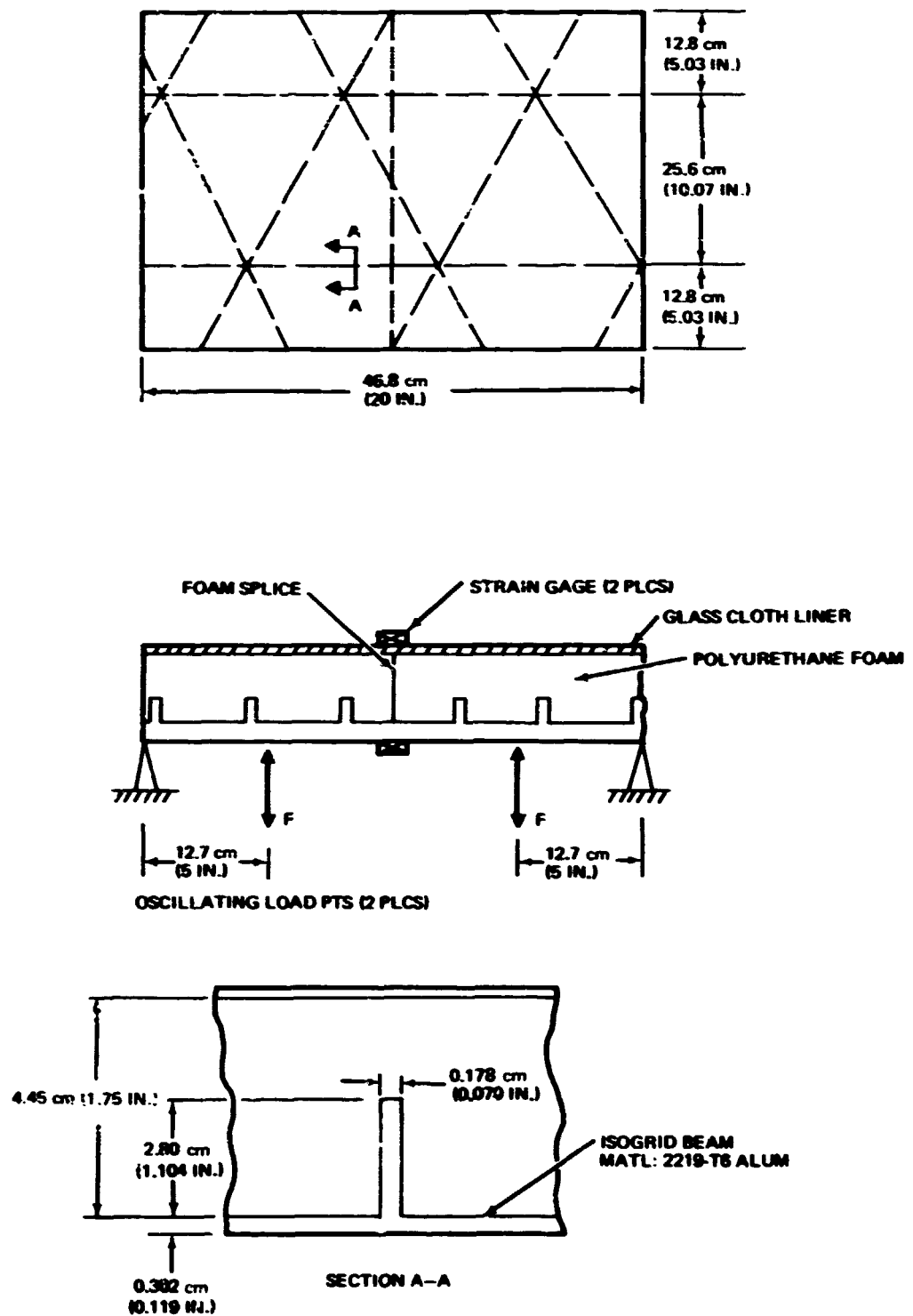


Figure 38. Schematic of Braided Composite Specimens

Table 23
INITIAL FATIGUE STRESS LEVELS IN THE GLASS
CLOTH LINER FOR THE BEAM BENDING
COUPON TESTS

	Temperature		Tensile Fatigue Stress		Fatigue Cycles
	C	(° F)	MN/m ²	(psi)	
Liftoff	-253	(-423)	8.27	(1,200)	36,000
Reentry	149	(300)	2.76	(410)	468,000

Table 24
PREDICTED INITIAL VALUES OF OSCILLATING
FORCE TO BE APPLIED TO FIRST
BEAM SPECIMENS

Temperature		F	
° C	(° F)	N	(lb)
-253	(-423)	507	114
150	(300)	397	69

NOTE: These values of F are to be applied at two locations on beams, 25% and 75% length of beam.

would provide an adequate factor of safety on life to account for fatigue scatter. The initial stress levels for the second specimen at temperature condition would be 50 percent higher than the failure level of the first specimen. Similarly, the initial stress levels for the third specimen would be 50 percent higher than the failure stress of the second specimen.

Two additional beam coupons would be tested (one at each temperature) at the failure level of the first specimen. A constant stress level would be applied until failure occurs. These tests should increase confidence in the application of Miner's Method to the construction of fatigue curves for this application.

A probable result of this testing is shown in Figure 39.

These data could then be used to construct a fatigue allowable curve by using Miner's Cumulative Damage Method, i.e., failure occurs when

$$\sum_{i=1}^k \eta_i / N_i = 1.0$$

η_i = number of applied load cycles at stress level i

N_i = allowable number of cycles at stress level i

k = number of different stress levels

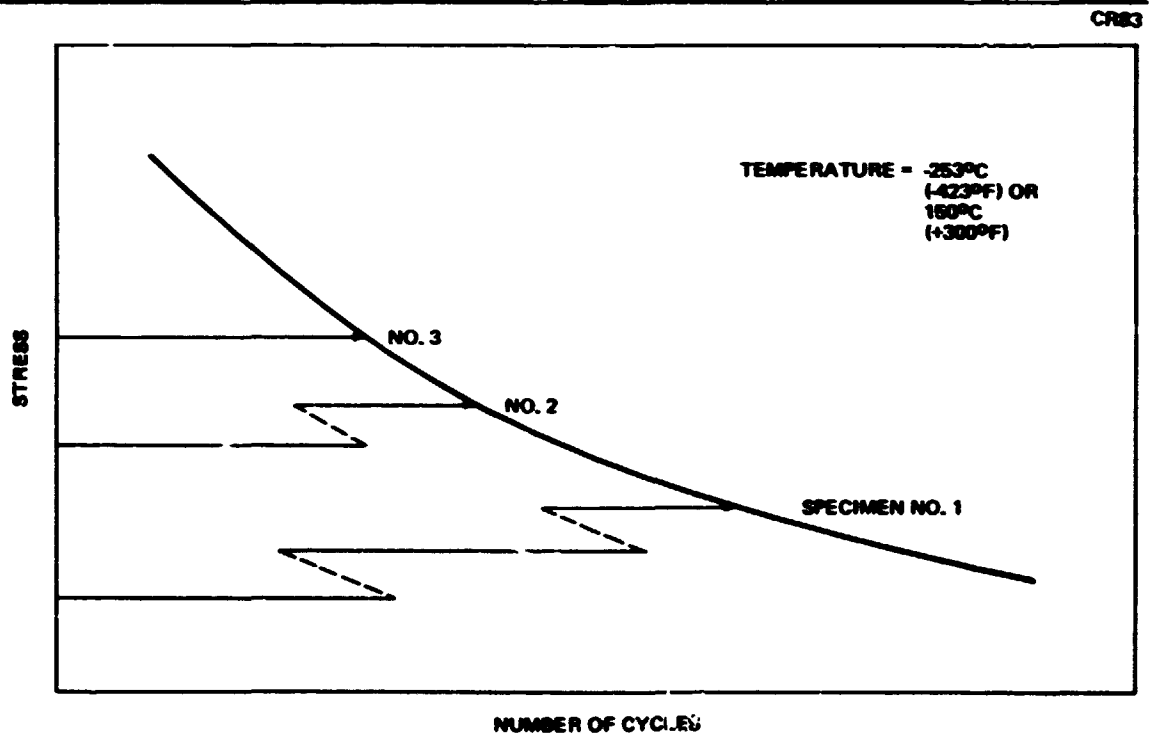


Figure 39. Fatigue Curve

An experimental SN curve is formed by fairing a best fit curve through the three data points while accounting for multiple load levels using Miner's Cumulative Damage Method.

Miner's Cumulative Damage Theory might also be used to construct a life cycle plot. This plot sums the fatigue damage occurring during liftoff, maximum dynamic pressure and reentry and relates the allowable life in lifeblocks to the ratio of applied loading over the predicted flight loading. An example of this type of curve is shown in Figure 40.

Acoustic tests would be performed on two panel specimens (Figure 41) to verify the capability of the advanced insulation and to confirm the validity of the fatigue allowables generated from the beam coupon tests. One specimen would be tested at each of the two extreme temperature conditions. The acoustic test levels for both specimens are shown in Table 25. Strain gages would be used to record the dynamic stresses in the aluminum panel and glass cloth liner during the tests. Testing would continue until failure or until an adequate sound level resistance was demonstrated.

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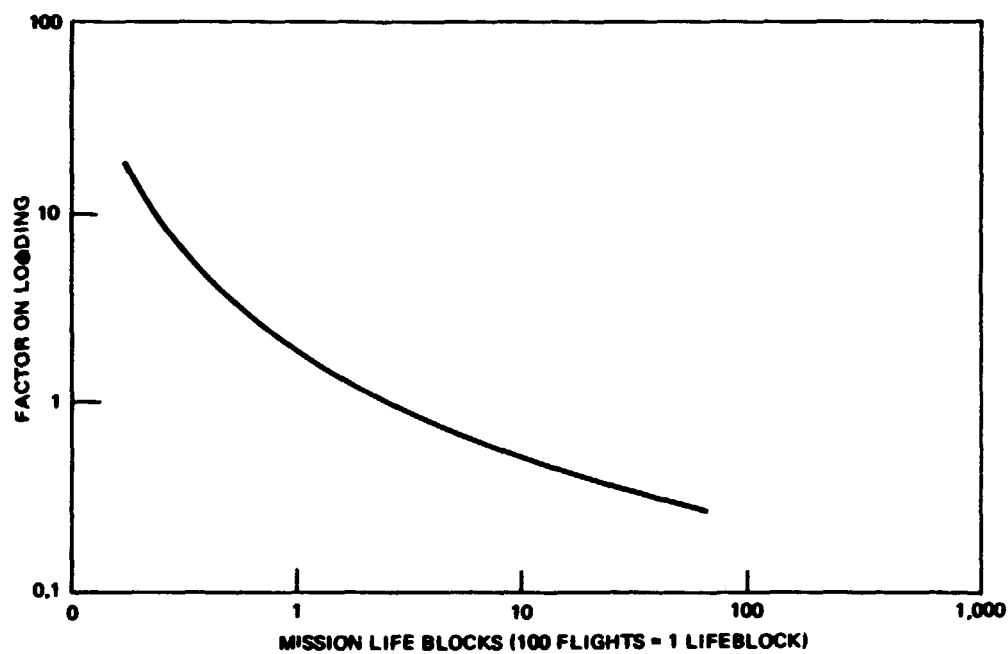


Figure 40. Mission Life Blocks

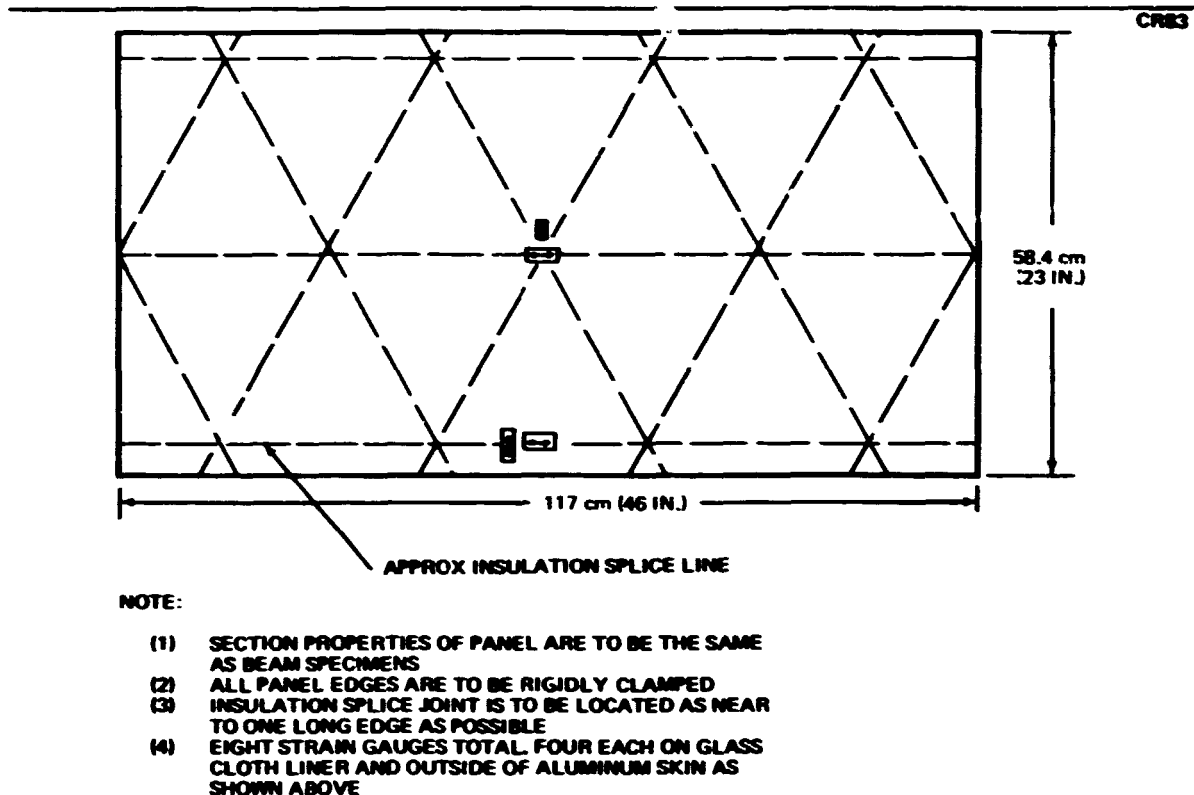


Figure 41. Panel Configuration for Acoustic Tests

2.5 PANEL JOINTS (TASK 5)

The primary objective of this task was to evaluate analytically the effect of various panel joint configurations on the thermal performance and structural integrity of the insulation system. A secondary objective of this task was to assess the effect of various panel joint configurations on vehicle insulation system repair techniques.

2.5.1 Thermal Analyses

It has been speculated that heat transfer along a joint may be an important consideration in determining the effective thermal conductivity of a foam insulation. As a result, an analysis was performed in an attempt to assess the magnitude of the contribution of different joint configurations to the overall heat transfer through the shuttle internal insulation system.

Assuming a shuttle related structural design, as shown in Figure 42, it can be seen that a multiplicity of variations can exist in the basic joint configurations, both from a dimensional sense and in the shape of the configurations.

Table 25
ACOUSTIC LEVELS FOR PANEL TESTS

1.3 Octave Band Center Frequency (Hz)	Level (dB) Temperature = -253°C (-423°F)	Level (dB) Temperature = 150°C (300°F)
5	119.0	114.5
6.3	120.5	116.0
8	122.0	117.5
10	124.0	119.5
12.5	125.5	121.0
16	127.0	122.5
20	128.5	124.0
25	130.5	126.0
31.5	132.0	127.5
40	133.5	129.0
50	135.0	130.5
63	137.0	132.5
80	138.5	134.0
100	140.0	135.5
125	140.5	136.0
160	141.0	136.5
200	141.0	136.5
250	141.0	136.5
315	141.0	136.5
400	141.0	136.5
500	140.5	136.0
630	140.0	135.5
800	139.0	134.5
1000	138.0	133.5
1250	137.0	132.5
1600	136.0	131.5
2000	135.0	130.5
2500	134.0	129.5
3150	133.0	128.5
4000	132.0	127.5
5000	131.0	126.5
6300	130.0	125.5
8000	129.0	124.5
10,000	128.0	123.5
Overall	151.5	147.0

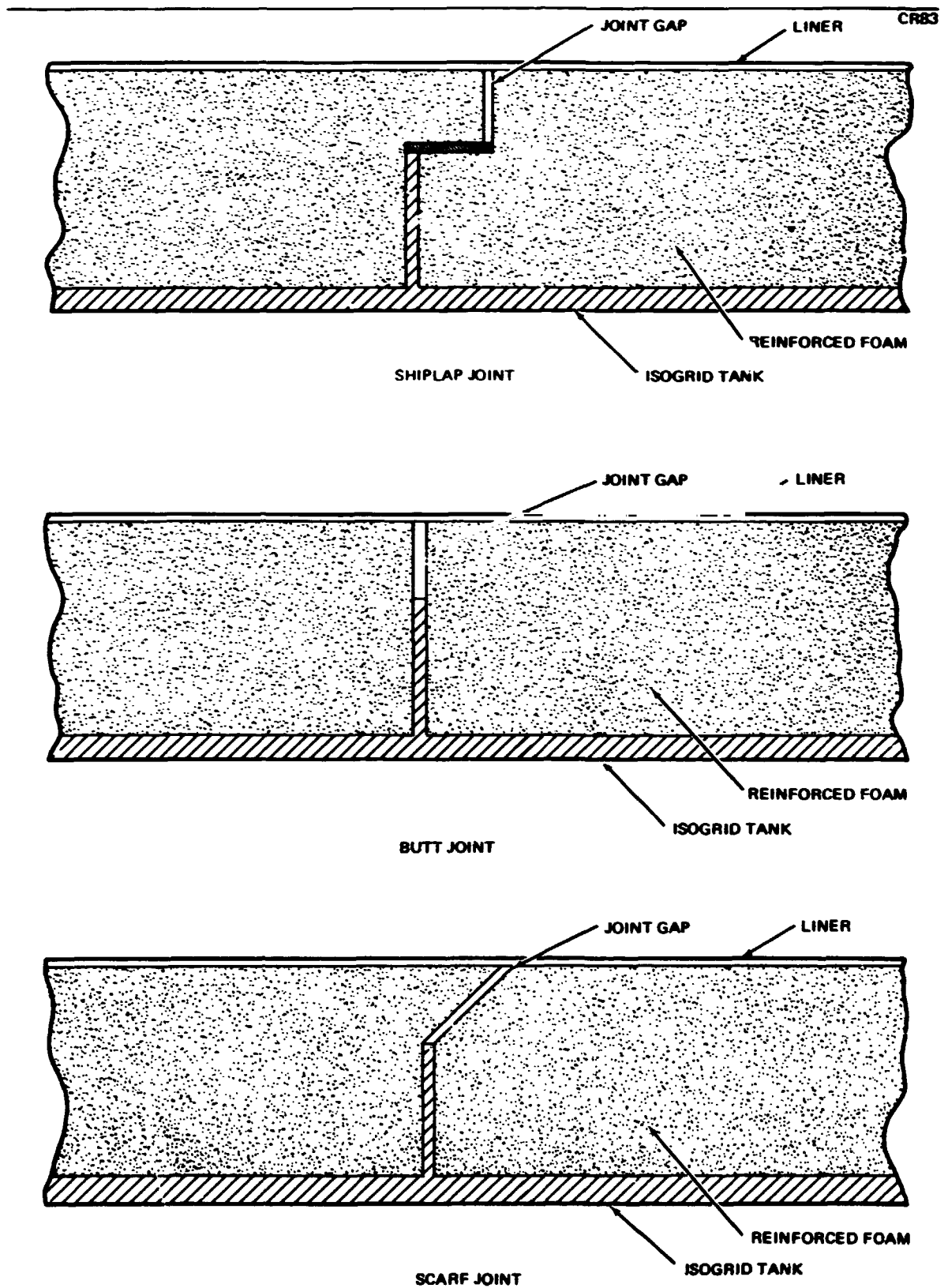


Figure 42. Joint Configurations

Two basic configurations were chosen for evaluation. These consisted of the butt joint and the shiplap joint. It was planned to evaluate these configurations with respect to a controlled area element without any joint by comparing the difference in heat transfer between the configurations.

Available data and those obtained from Saturn flights indicate that the thermal conductivity of the overall system is relatively close to the thermal conductivity of helium or hydrogen gas (Figure 43). This occurrence is due to permeation of the liner and resultant subsequent saturation of the insulation with gas.

The heat transfer modes which were considered to play a possible role in the heat transfer process within the foam insulation included conduction, convection, and radiation.

With the existence of an internal liner, the saturation with LH_2 of the insulation tiles and gaps in the joints of insulation tiles (in the case of

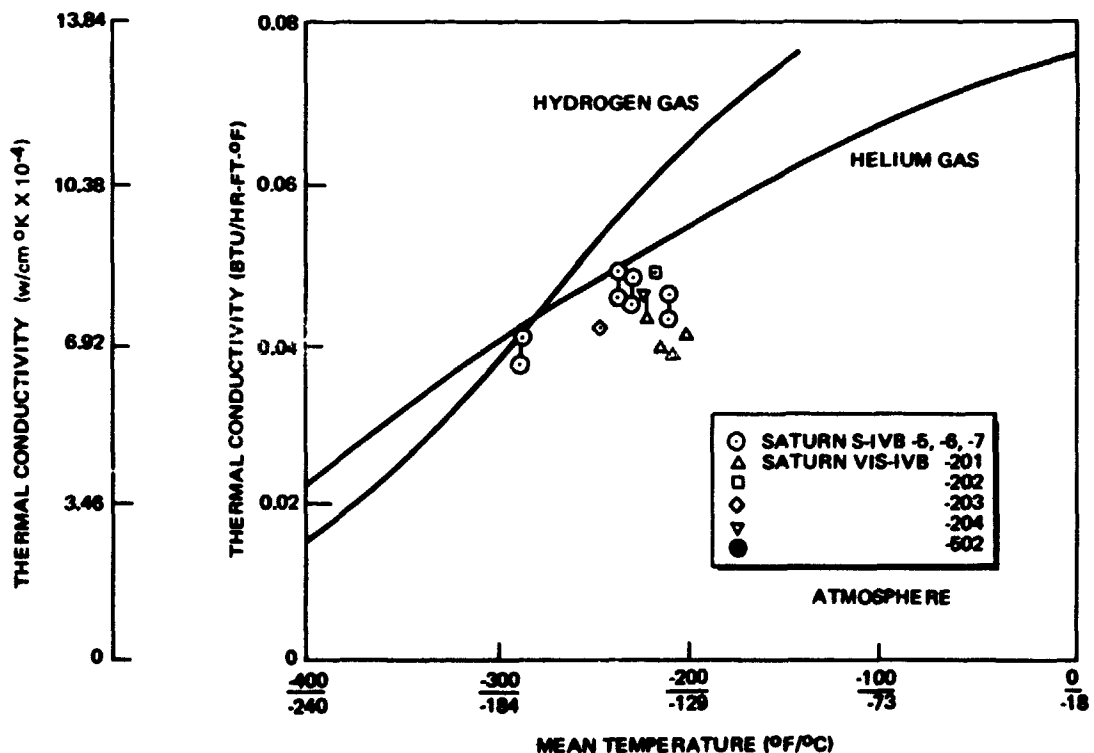


Figure 43. 3-D Reinforced Saturn Foam—Maximum Thermal Conductivity

unbonded joints) is precluded. However, physical gaps between insulation tiles will fill with hydrogen gas. As a result, the possibility of convective heat transfer occurring within such gaps was contemplated. A review of the parameters involved in the free convection process indicated that for spacings less than 0.32 cm (1/8 inch) in width, the heat transfer due to free convection is essentially nonexistent. In addition, the liner prevents LH_2 from contacting the wall, thereby precluding circulation due to boiling within the joint.

The only significant modes of heat transfer then are radiation and conduction.

The thermal analysis was carried out using the thermal properties of the Saturn 3-D foam (since more data on the S-IVB material were available and since it was assumed that the thermal properties of the two materials should be similar for the same density). The properties used are summarized in Table 26.

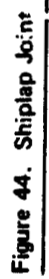
The basic thermal models used are shown in Figures 44 and 45. An area of 0.0516 m^2 (0.557 square feet) was assumed for each model. The models were constructed for the MDAC JA03 3-dimensional heat transfer program. Boundary temperatures of 149°C (300°F) and -253°C (-423°F) were assumed for the wall and the liner, respectively. For the case of an insulation tile without a joint, the steady state heat transfer was shown to be approximately 86.8 watt/m^2 ($276 \text{ Btu/ft}^2\text{-hr}$) for a thickness of 3.8 cm (1.5 inches) and a temperature differential of 384°C (723°F).

An attempt was made to determine the effect of radiation across joint gaps. Gaps were varied from 0.076 cm (0.03 inch) to 0.32 cm (0.125 inch) in width. Radiation view factors and interchange factors were determined and used in both joint configuration models in order to determine the difference in radiation between these configurations. Typical values are plotted as a function of gap width in Figure 46.

It can be seen from this figure that the radiative contribution rises at a rapid rate as the gap width increases. However, the absolute level of heat transfer due to this mode is more than 3 orders of magnitude lower than the overall heat transfer through the tiles, and for this reason was considered to be insignificant.

Table 26
3-D FOAM THERMOPHYSICAL PROPERTIES

Temperature		Density		Specific Heat		Conductivity	
°C	(°F)	kg/m ³	(lb/ft ³)	joule/kg·°C	(Btu/lb·°F)	joule/m·sec·°C	(Btu/ft·hr·°F)
-253	(-423)	48	(3.0)	314	(0.075)	0.024	(0.014)
-240	(-400)			627	(0.15)	0.036	(0.021)
-229	(-380)			635	(0.152)	0.045	(0.0258)
-218	(-360)			644	(0.154)	0.052	(0.03)
-207	(-340)			652	(0.156)	0.059	(0.034)
-196	(-320)			660	(0.158)	0.065	(0.0375)
-184	(-300)			670	(0.16)	0.070	(0.0402)
-173	(-280)			682	(0.163)	0.075	(0.043)
-162	(-260)			694	(0.166)	0.080	(0.046)
-151	(-240)			706	(0.169)	0.083	(0.048)
-140	(-220)			719	(0.172)	0.085	(0.049)
-129	(-200)			736	(0.176)	0.087	(0.05)
-112	(-170)			765	(0.183)	0.088	(0.051)
-93	(-135)			833	(0.192)	0.089	(0.0515)
-51	(-60)			904	(0.216)	0.093	(0.0535)
-23	(-10)			987	(0.236)	0.094	(0.054)
32	(90)	48	(3.0)	1,204	(0.288)	0.094	(0.0545)
93	(200)			1,204	(0.288)	0.095	(0.055)
149	(300)			1,204	(0.288)	0.095	(0.055)



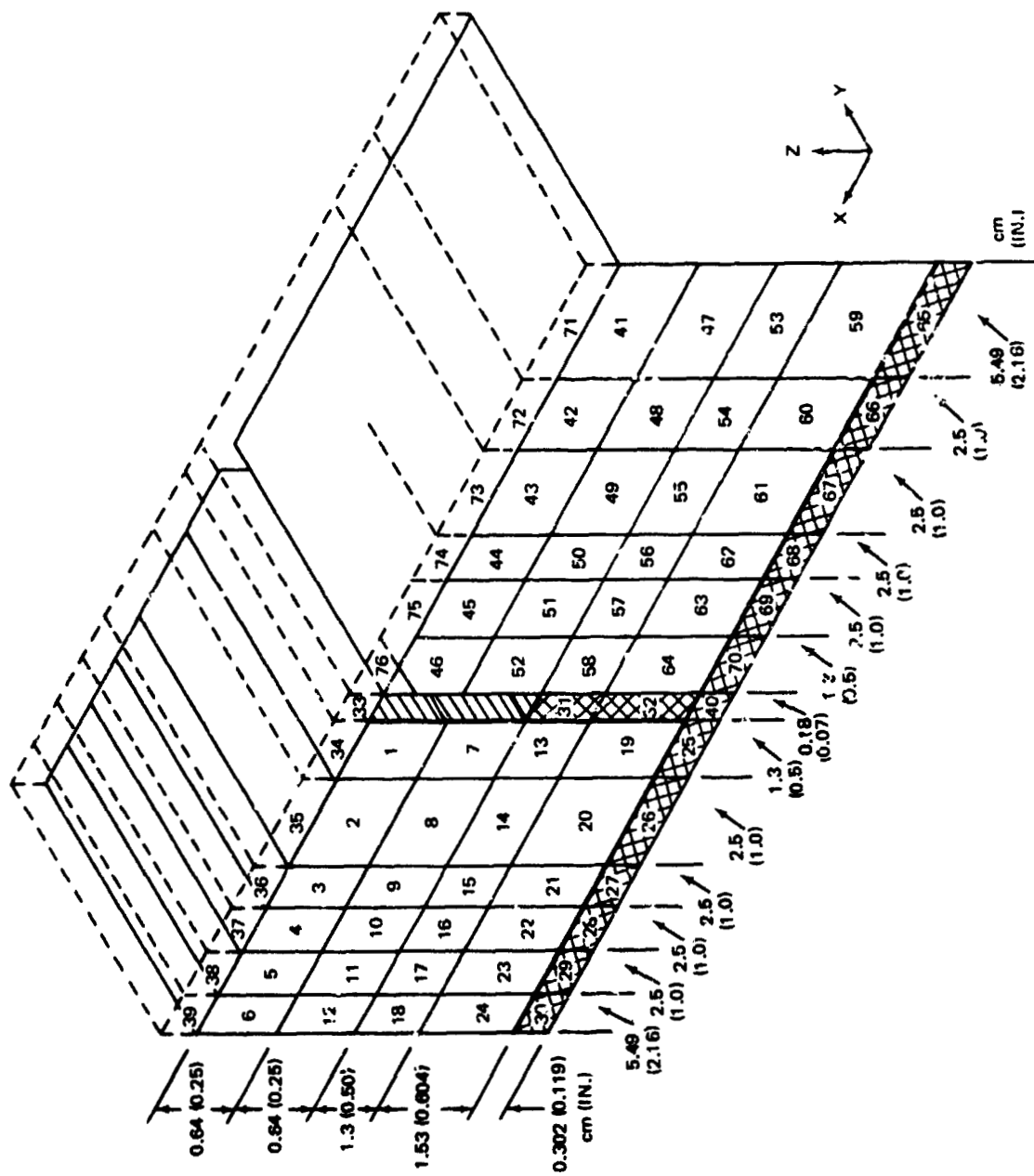


Figure 45. Butt Joint

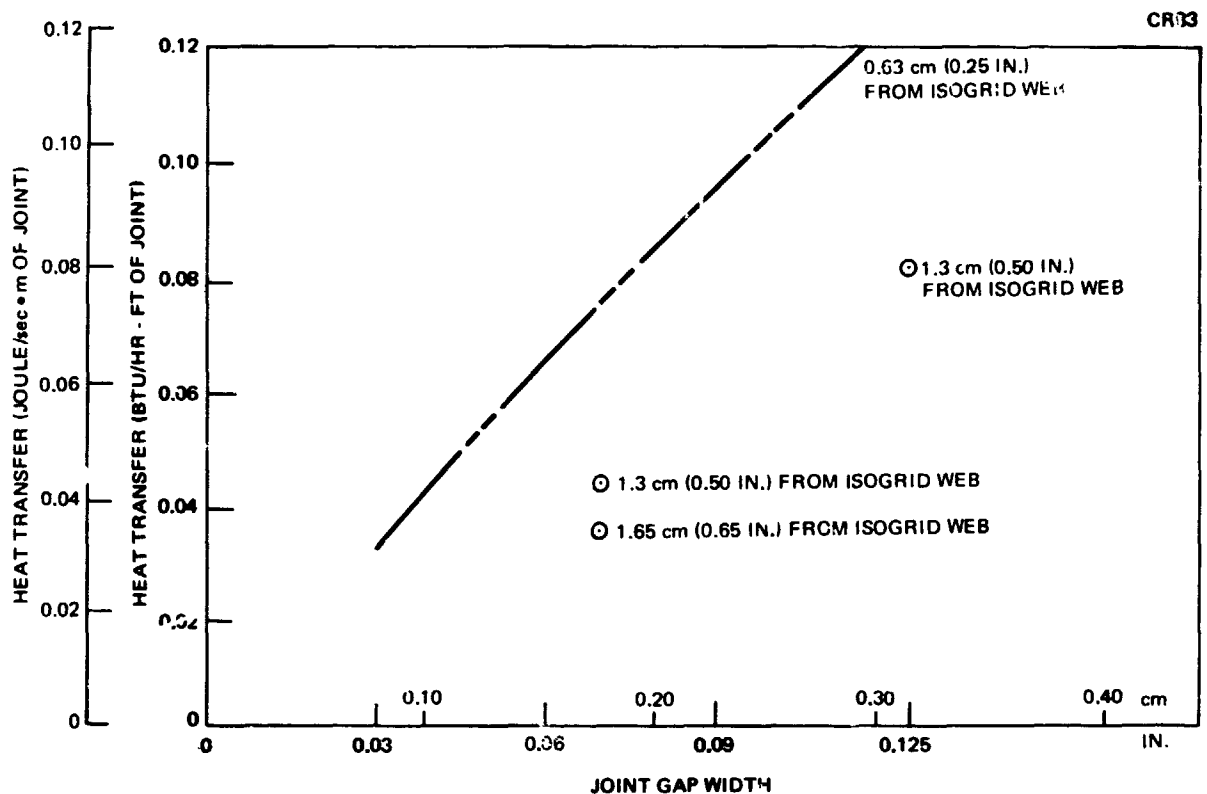


Figure 46. Radiation Heat Transfer Contribution. Shiplap Joint

The effect of distance between the joint and the isogrid web is shown for several gap width/spacing distance combinations in Figure 46. As would normally be expected, there is a reduction in the heat transfer with an increase in distance between the joint and the web. This is due primarily to decreasing temperatures within the foam with increasing distance from the web.

A typical temperature distribution within the foam is shown in Figure 47. This distribution indicates that heat transfer will be greatest in the vicinity of the isogrid web because of the high temperature of the aluminum web and its penetration through a major part of the total foam thickness. It should be noted, however, that the temperature distribution within the foam is due primarily to the presence of the aluminum web, not to the joint, and that the distribution is essentially unchanged regardless of the position of the joint with respect to the web.

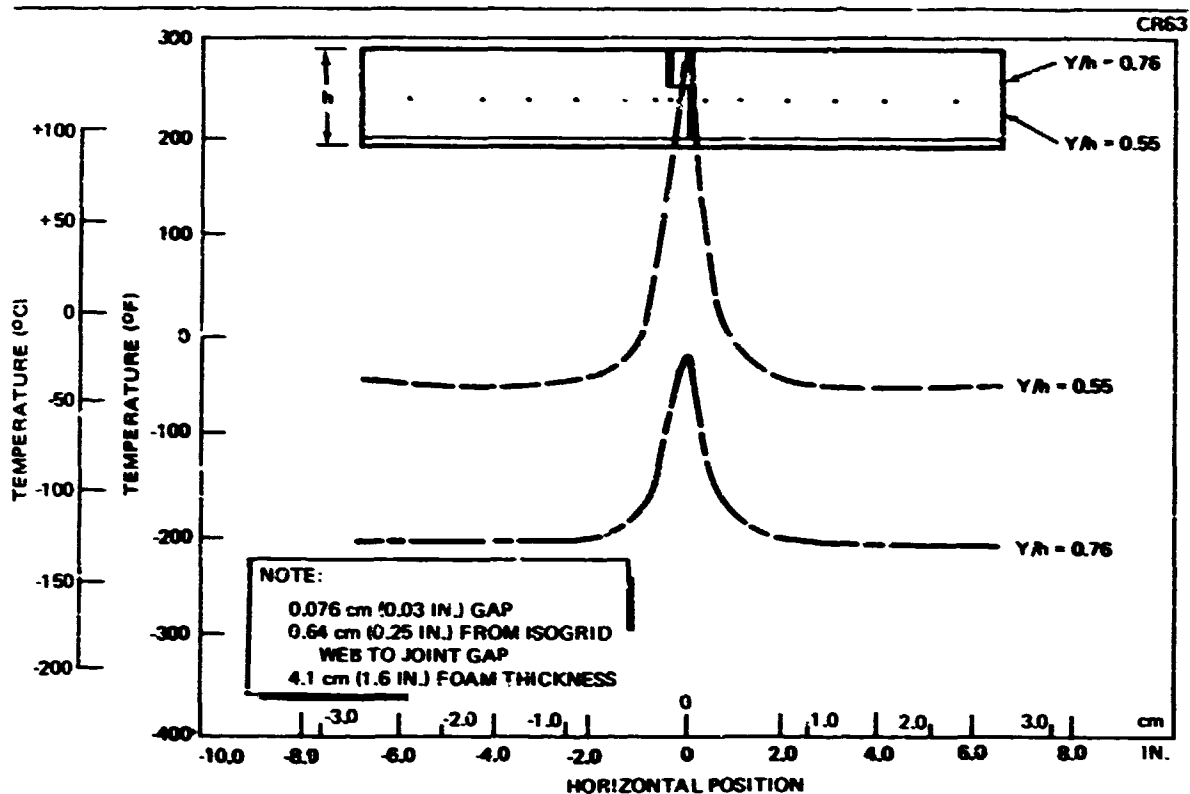


Figure 47. Horizontal Temperature Distribution, Shiplap Joint

In summary, the analysis performed on joints in 3-dimensional foams indicated that, in view of the fact that the foam will take on a thermal conductivity approaching that of hydrogen, there will be little noticeable difference in conduction heat transfer through insulation tiles regardless of joint configuration. Radiated heat transfer across gaps, in the case where gaps exist, will contribute an insignificant amount of heat transfer to the overall heat transfer through the system.

It was concluded that the choice of joint configuration could be made on the basis of structural and manufacturing requirements rather than on the basis of achieving optimum thermal performance.

2.5.2 Structural Evaluation of Panel Joint Configurations

The baseline shuttle LH_2 tank structural configuration selected for the study is shown in Figure 27, Subsection 2.4. The tank is an internally stiffened integral isogrid shell with approximately 1.3 cm (0.5 inches) of insulation over the stiffener. The insulation panels are equilateral triangular blocks 29.2 cm (11.5 inches) on the side by 4.1 cm (1.6 inches) thick. Depending

upon the panel joint configuration, the isogrid rib would be left uncovered, covered by a foam lap, or covered with a filler material. The panels are bonded to the aluminum skin, but may or may not be bonded to adjoining panels.

Three insulation panel joint configurations, a butt joint, a scarf joint, and the currently used overlap joint, were evaluated to determine the structural response of the tank liner and foam insulation. The configuration analysis took into account manufacturability as well as the effects of pressure and thermal loads. The effect of bond material between adjoining panels was also evaluated. Figure 42 shows the three joint configurations considered.

Unfilled gaps between the foam panels result in stress concentrations in the foam-liner bond line. When the gap is sufficiently wide, the stress concentration initiates a peel type of failure. Hence, the relative magnitude of the stress concentration will be a factor in selecting a joint configuration. The analysis considered a critical condition in which the tank pressure is suddenly reduced, creating a momentary pressure differential across the liner of 0.21 MN/m^2 (30 psi). This pressure tends to blow the liner away from the insulation resulting in tensile bond stresses perpendicular to the bond line. (See also Section 2.3.8).

The analysis model was a beam on a discontinuous elastic foundation loaded by a uniform lateral pressure, Figure 48. Taking a 2.5 cm (one inch) wide strip of liner as the beam yielded

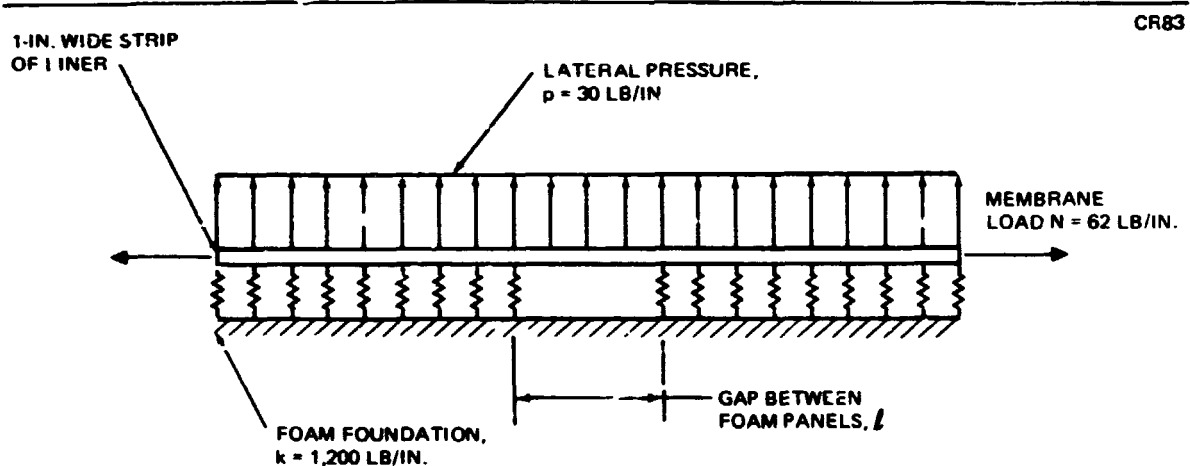


Figure 48. Beam Column Analysis Model

$$\text{Beam Area} = (0.0045 \text{ in.})(1.0 \text{ in.}) = 4.5 \times 10^{-3} \text{ in.}^2 \\ (2.9 \times 10^{-6} \text{ m}^2)$$

$$\text{Beam Moment of Inertia} = (1.0 \text{ in.})(0.0045 \text{ in.})^3/12 \\ = 7.6 \times 10^{-9} \text{ in.}^4 \quad (3.2 \times 10^{-15} \text{ m}^4)$$

$$\text{Foundation Spring Constant} = \frac{E_{\text{foam}}}{d_{\text{foam}}} = \frac{2100 \text{ lb}}{1.75 \text{ in.}} = 1200 \text{ lb/in.} (21.0 \text{ N/m})$$

$$\text{Membrane Load} = [\alpha \Delta T_{\text{liner}} - \alpha \Delta T_{\text{AL}}] E t_{\text{liner}} \\ = [(9.5^\circ \text{F}^{-1})(423^\circ \text{F}) - (12^\circ \text{F}^{-1})(300^\circ \text{F})] \\ (1.8 \text{ lb/in.}^2)(0.0045 \text{ in.}) \\ = 62 \text{ lb/in.} (1.08 \text{ N/m})$$

A standard MDC computer program for analyzing beam columns on elastic foundations, MW03, was used to compute deflections and stresses along the beam. Figure 49 is a typical plot of deflections due to a gap of width, l .

The bending stress in the liner is small, on the order of 41 MN/m^2 (6,000 psi). However the tensile stresses in the bond at the edge of the foundation become quite large, compared to the ultimate allowable bond stress, as the unsupported length of liner is increased. These stresses are shown in Figure 50 for two extreme axial load cases. One case is no axial load in the liner, i.e., no temperature differential through the insulation,

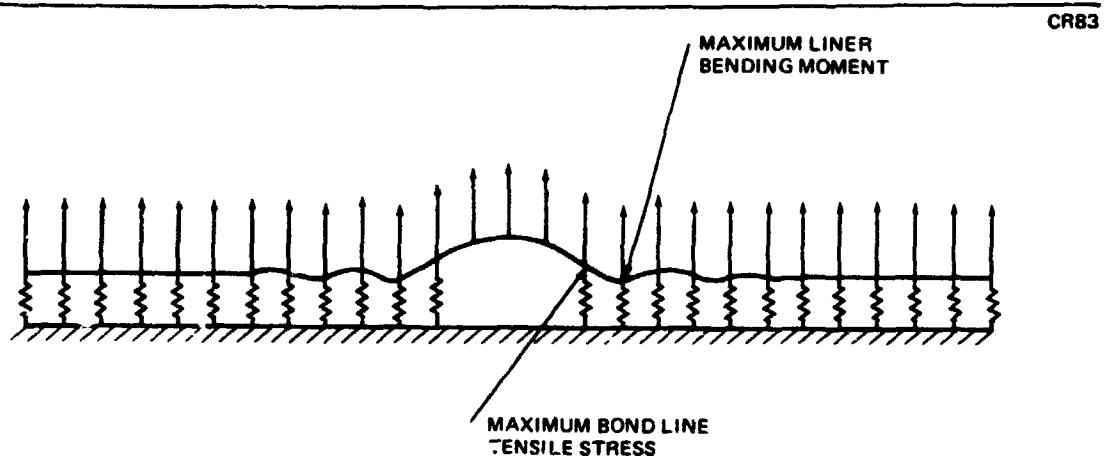


Figure 49. Typical Deflected Shape of Liner

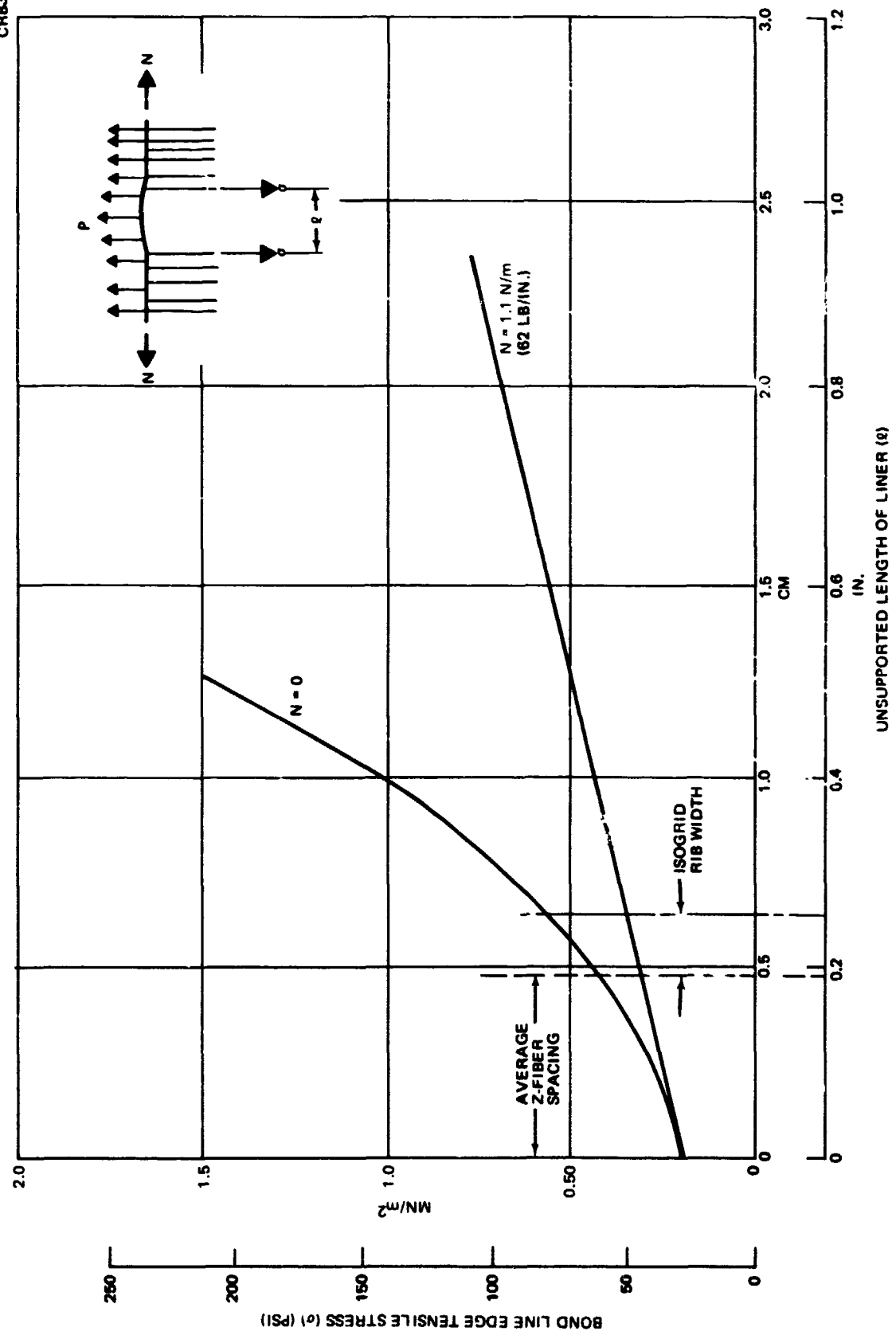


Figure 50. Bond Line Tensile Stress at Edge of Unsupported Liner

the other assumes the liner is at -253°C (-423°F) while the aluminum is at 149°C ($+300^{\circ}\text{F}$) resulting in a load of 1.1 N/m (62 pounds/inch) in the liner.

Two conclusions can be drawn. First, the smaller the gap width, the less the chance of a peel failure. Second, gap widths of the same size as the isogrid rib appear to pose no structural problems from liner debonding (as indicated by Saturn experience) or from tensile failure of the liner.

Another analysis was conducted to determine the structural response of the foam insulation in the vicinity of the joint to pressure and thermal loads. To determine whether the bonding together of adjacent panels is structurally advantageous, each configuration was analyzed with and without bond material between the adjoining panels.

The SAAS III⁸ Finite Element Computer Program was used to determine displacements, stresses, and strains near the joint. The program represented the cylinder wall, foam and liner as a system of solid ring elements with quadrilateral cross sections. Consequently, geometries of complex shape and of several different orthotropic materials could be analyzed.

Input to the program included temperature distributions from the JA03 thermal analysis program (Paragraph 2.5.1); material properties as a function of temperature for the aluminum, foam, liner, and bond material; pressure loads and boundary conditions. By assuming a plane strain boundary condition in the axial direction, a short segment of the cylinder could be modeled as shown in Figure 51. The differential pressure across the aluminum wall was 0.21 MN/m^2 (30 psi). A gap width of 0.18 m (0.070 inches), the same as the isogrid rib width, was assumed.

The potential failure modes for all joint configurations are a tensile bond failure between the aluminum and the foam and a tensile rupture of the x-y fibers just under the liner. The analysis showed these potential failure modes are present whether a joint exists or not and are not seriously affected by any of the joint configurations. The most critical stress was a

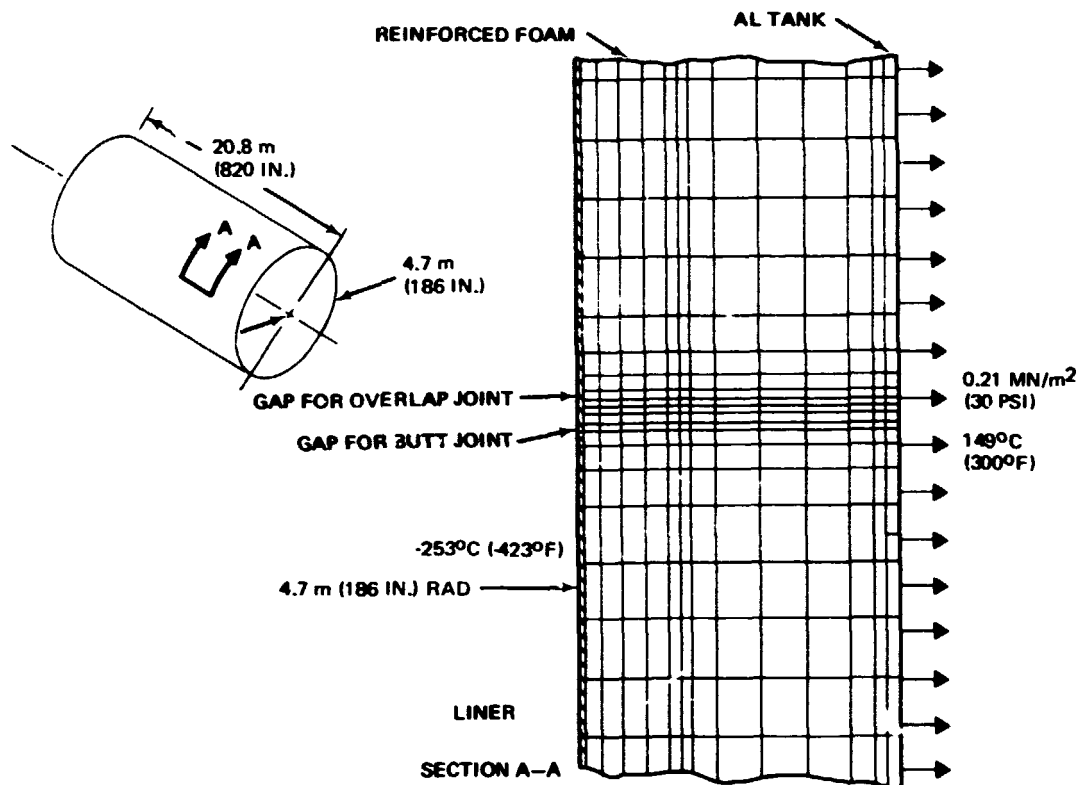


Figure 51 Finite Element Model

tensile stress in the foam just beneath the liner of 0.104 MN/m^2 (15 psi) at -253°C (-423°F) in the meridional direction. Compared to an allowable stress of 1.14 MN/m^2 (165 psi) (developed by an x-y joint), a high margin of safety results.

In conclusion, the selection of a joint design did not appear to be strongly influenced by structural considerations. The analysis showed that structurally, it is wise to maintain a reasonably small gap between adjacent panels; however, gaps the same size as the isogrid ribs did not appear to be detrimental and need not be filled.

2.5.3 Influence on Liner Strength by Filled Joints in 3D Foam

The relationship between the Epon 828/CL bonded glass fabric liner and the joint in 3D foam was also explored by coupon testing in LN_2 (-196°C ; -320°F). The object of these tests was to determine the crack sensitivity of the liner along the edges of a rigid gap filled joint when subjected to cryogenic contraction load, and to determine if the liner would develop its expected cryogenic rupture strength when bonded across a rigid gap filled joint.

A standard design tensile bond strength specimen was prepared except the length of 3D foam was extended to 30 cm (12 inches) so that a joint at the midsection 15 cm (6 inches) from each end would be isolated from the loading blocks. This specimen design is presented in Figure 52 showing the butt joint and shiplap joint configurations tested. The 116 glass fabric/Epon 828/CL liner was wrapped completely around 2 sides and the ends of the blocks and bonded in place across the two opposite edges of the joint in the 3D foam. The test fixture and procedure for LN_2 contraction test is given in Figure 53. The joints in the 3D foam were filled with Lefkowied 211A/LZ for one series of tests and filled with a syntactic foam mixture for another series of tests. The syntactic foam consisted of the following formulation:

- 70 parts Epon 828/CL resin
- 18 parts phenolic microballoons
- 12 parts glass milled fibers

The syntactic foam gap filler handled as a thixotropic paste and when cured the density measured 480 kg/m^3 (30 PCF).

2.5.3.1 Results of LN_2 Contraction Test

The load developed through contraction of these specimens when immersed in LN_2 was approximately 890 N (200 pounds) and represented the combined contraction effects of liner (10 cm; 4 inches in width) and the 3D foam ($2.6 \times 10^{-3} \text{ m}^2$; 4 square inches in area). At this point no cracks were evident in the liner or in the gap filled joint. After rupturing the specimen in LN_2 a myriad of resin craze type cracks were visible in the liner surface but were not influenced by the joint in the 3D foam. The photograph presented in Figure 54 shows these craze marks are still visible at ambient temperature but are not as pronounced as when observed while still cold. The rupture strength in LN_2 of approximately 4,450 N (1,000 pounds) as shown in Figure 55 was lower than expected. The 10 cm (4 inch) width of liner should have ruptured in the 6,230 N (1,400 pound) range as indicated by the dotted line projection, however, the sharp corner of the metal attach block ripped the liner and terminated the test.

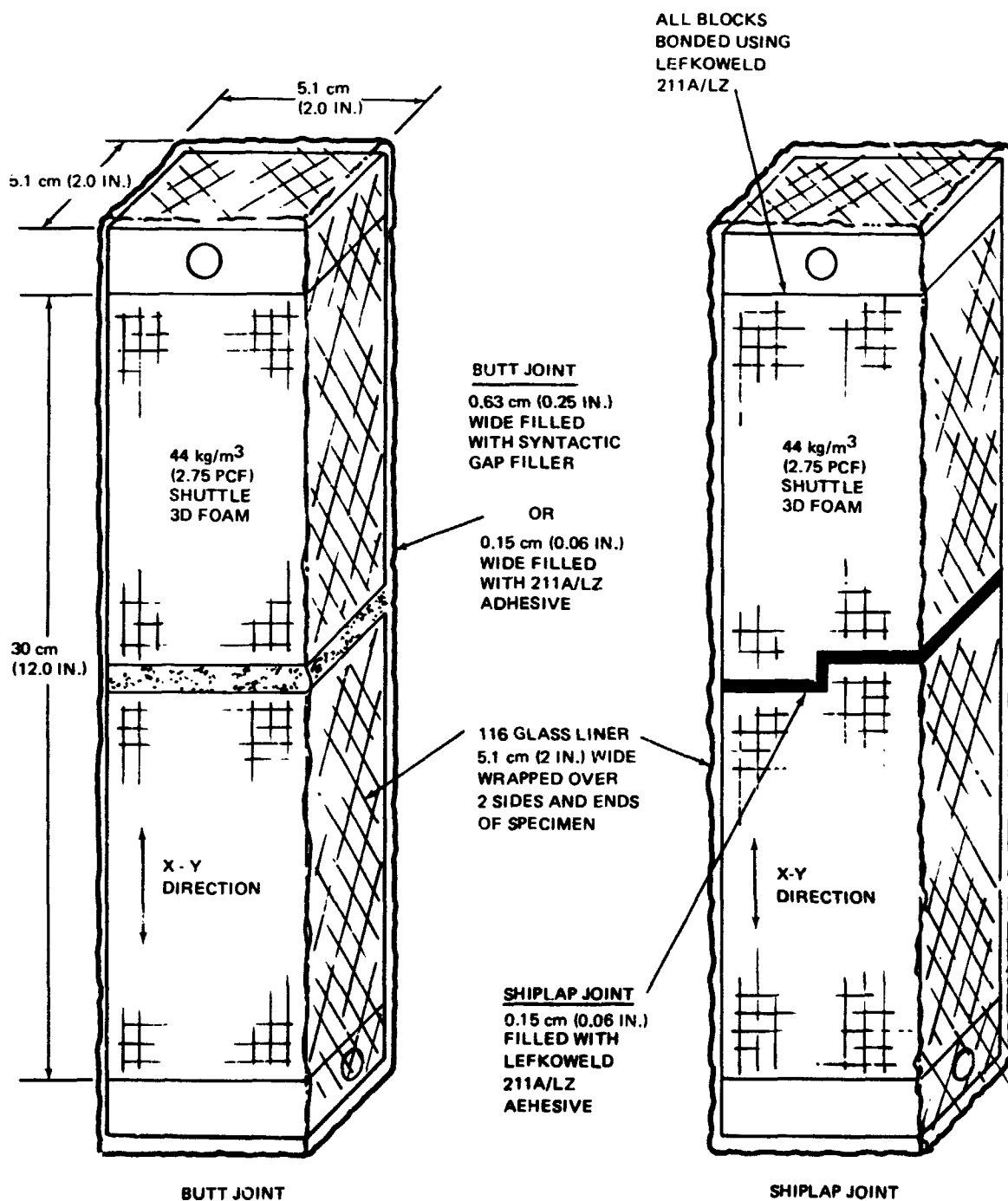


Figure 52. Joint Test Configuration 3D Foam

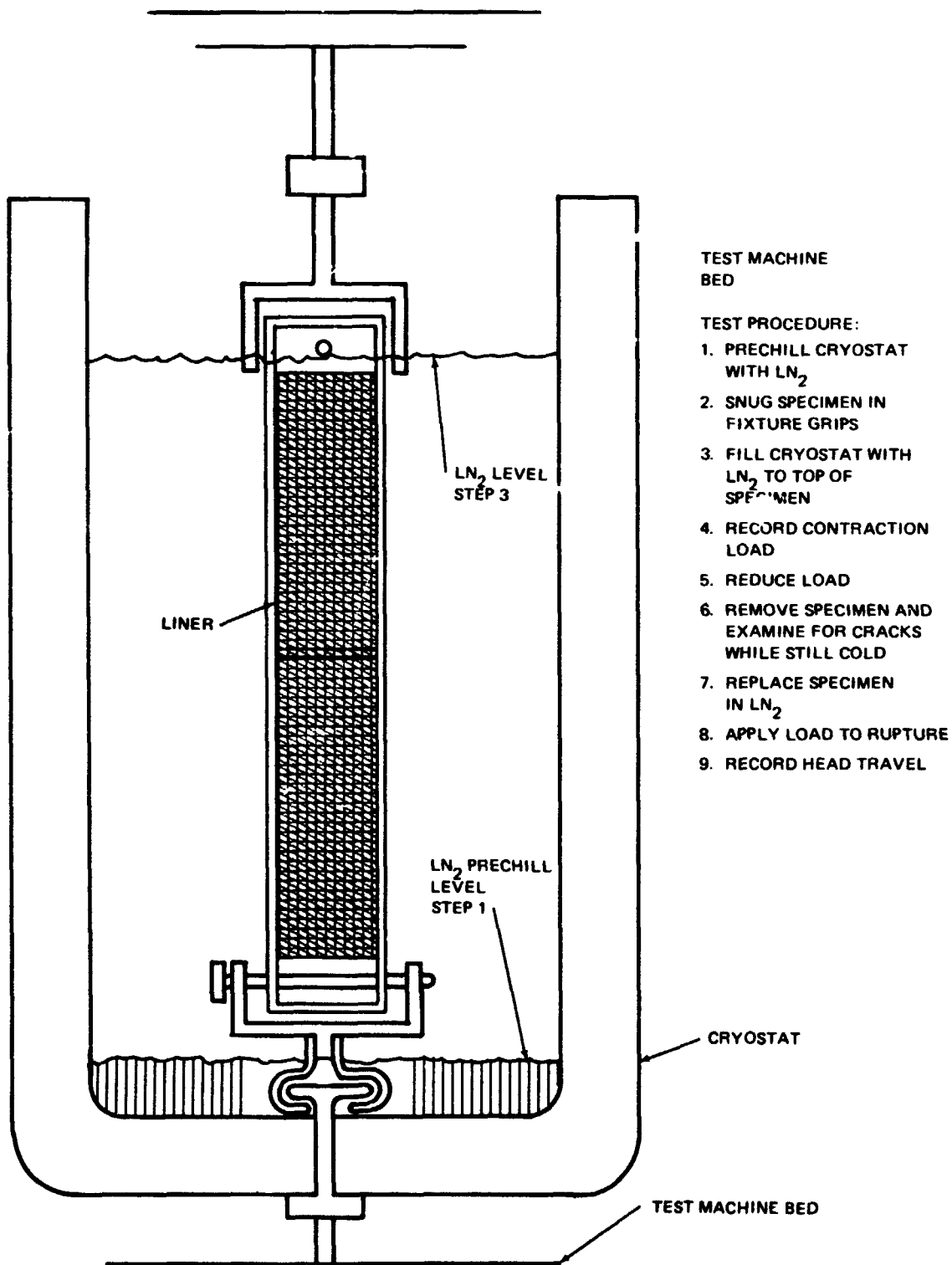


Figure 53. Test Setup for Joint Contraction 3D Foam

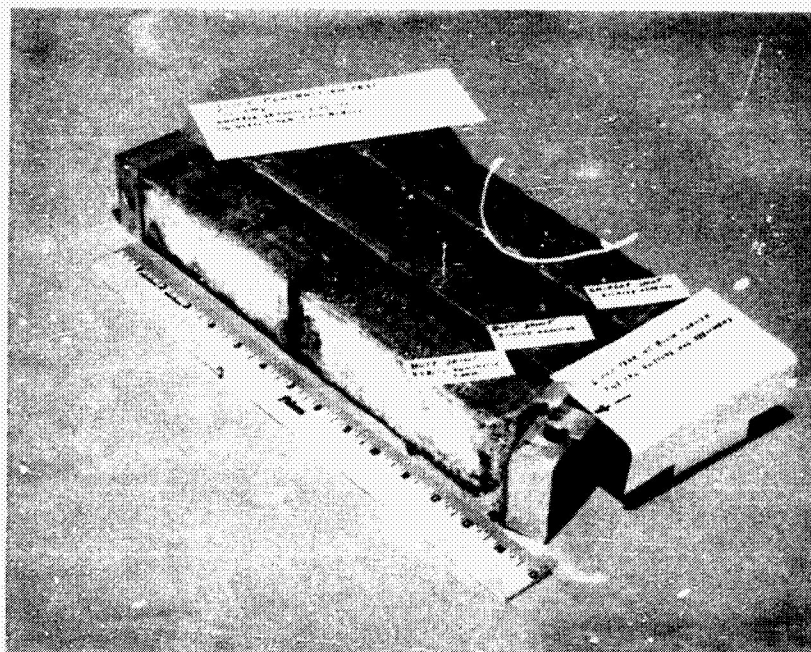


Figure 54. Joint Contraction Specimens After LN₂ Test

The results of these tests lend confidence to the structural analysis showing that no significant difference exists between butt joints and ship lap joints in the 3D foam. Also, there appeared to be no deleterious effect of the rigid gap filled joint on the glass liner. Either Lefkowied 211A/LZ or the synthetic foam using Epon 828/CL binder are recommended for use as a gap filler for 3D foam joints as would be accomplished during insulation bonding operations.

2.6 INSULATION REPAIR (TASK 6)

The objective of this task was to evaluate repair techniques for damaged or improperly installed insulation. Saturn S-IVB insulation repair techniques were used as a point of departure. Repairs considered were:

- A. Delaminated liner (blisters)
- B. Delaminated liner with torn fabric
- C. Crushed or damaged 3D foam and liner
- D. Large area of crushed or damaged foam and liner, including debonding of the 3D foam from the tank wall.

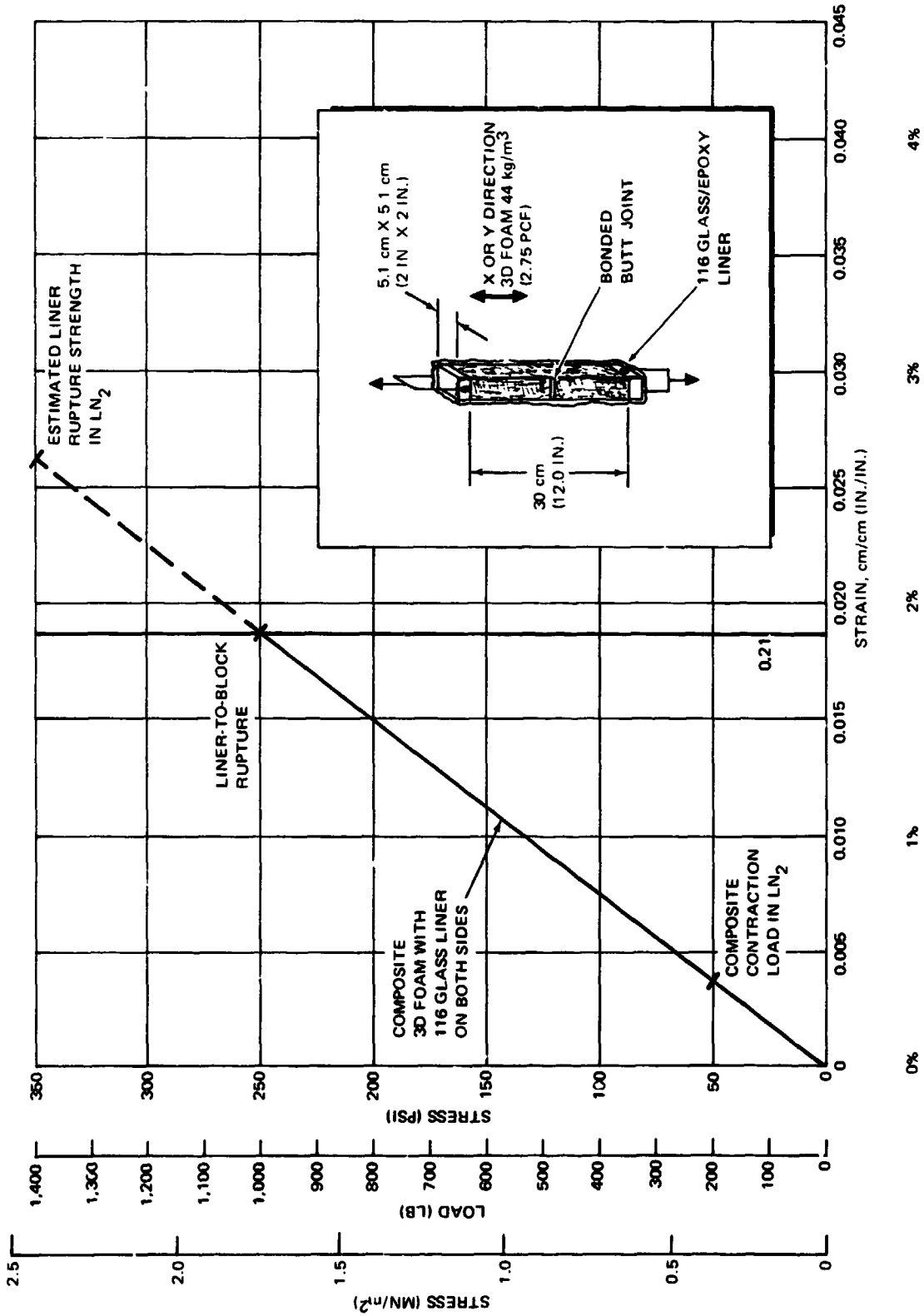


Figure 55. LN₂ Contraction Tests BX-251A-3D Bonded Joint

Using the principle that repaired areas of insulation must develop the same strength and functional efficiency as the parent material, the adhesive used in evaluation of repair techniques was Lefkowitz 211A/LZ (Epon 828/CL was used for liner repair).

The predominant number of repairs is of the small type, less than 4 square inches in area. These small areas of damage are induced during the insulation bonding operation or during instrument installation in the field. These repairs should be accomplished without the use of vacuum bag pressure or other equipment that is awkward to handle while the tank is in the vertical position or being rotated for assembly of instruments outside or inside the tank. The typical repair procedure applied to small damaged areas is described in Figures 56 and 57.

This basic repair method required resolution of several problem areas:

- A. Bond strength of "new" 3D foam plug to "old" adhesive layer.
- B. The only pressure applied during the warm cure cycle is developed through the compression of the sponge 40 kg/m^3 (2.5 PCF) polyurethane pad.
- C. The tape must hold tight to the surrounding liner against the back pressure of the compressed sponge during the 52°C (125°F) warm cure cycle.
- D. The method of applying heat while conducting field repairs must conform with safety requirements and be portable.

The test program to resolve these problem areas started with bond strength evaluations of double thickness 211A/LZ adhesive. Existing panels, $15 \times 30 \text{ cm}$ ($6 \times 12 \text{ inches}$) in area with 3D foam bonded to anodized 2219-T87 were used for these tests. The 3D foam was scraped off using a sharp acrylic blade leaving the "old" adhesive intact on the aluminum surface. Another slice of 3D foam was then coated with adhesive to obtain a layer weighing 0.75 kg/m^2 (70 grams per square foot). This excess adhesive weight [normal is 0.49 kg/m^2 (45 gms/ft.²)] was in keeping with the practice expected for bonding to rough irregular surfaces. A 5 cm (2 inch) thick pad of polyurethane 40 kg/m^3 (2.5 PCF) sponge was placed over the 3D foam and taped in place to compress the sponge to one inch thickness.

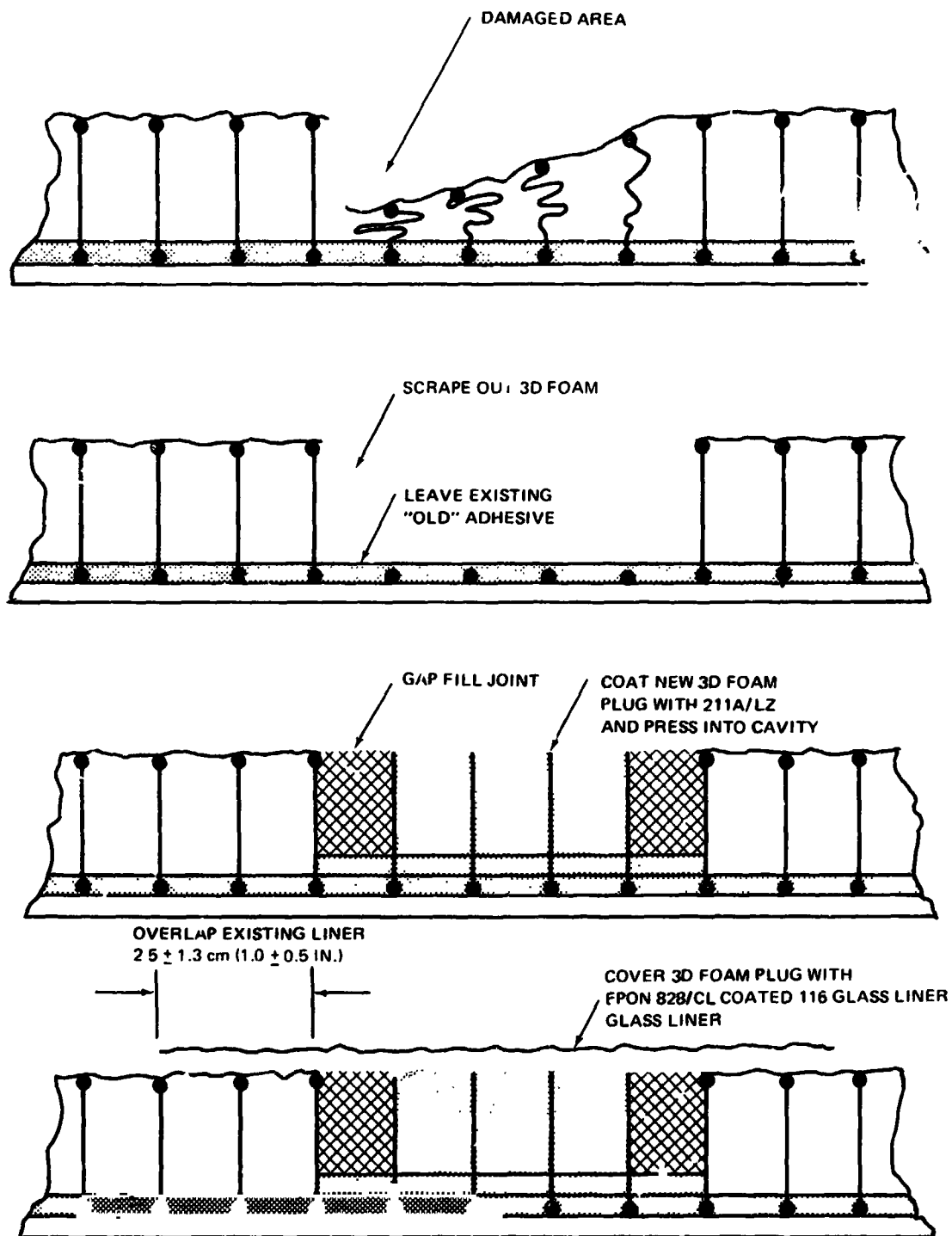
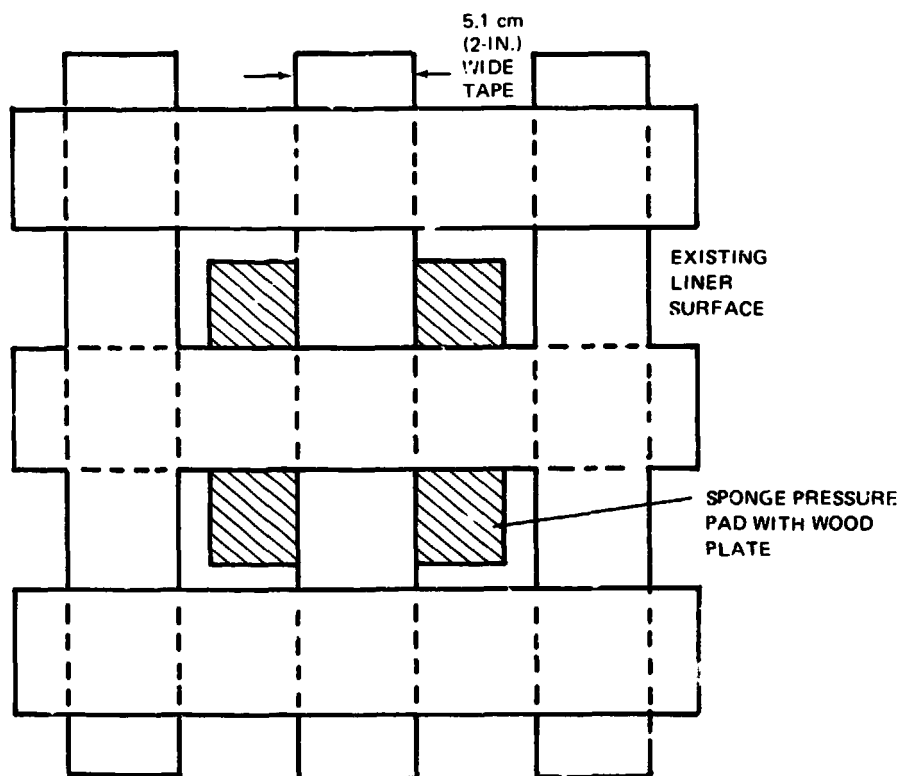
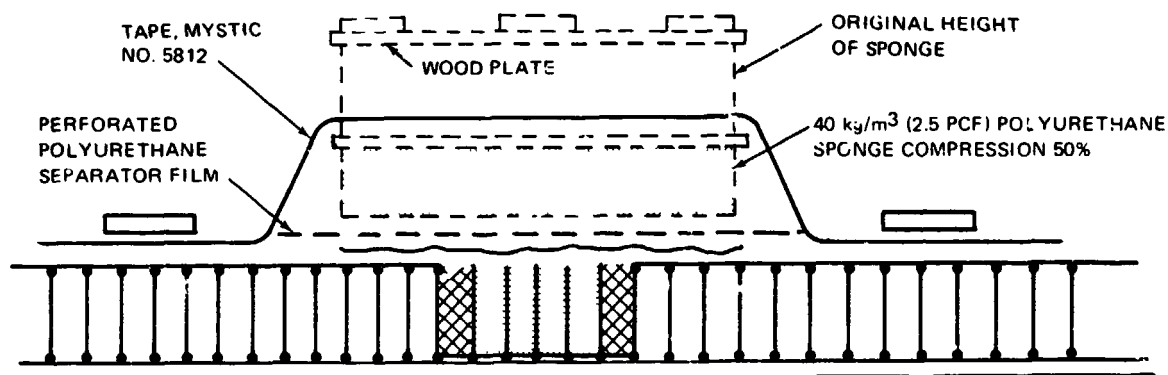


Figure 56. Typical Repair Procedure – Small Damaged Area



APPLY LOCALIZED HEAT TO TANK WALL FROM OUTSIDE SURFACE OF TANK WHILE MONITORING THE TEMPERATURE AT BOTH LINER AND TANK WALL SURFACES.

REMOVE PRESSURE PAD AND POST-CURE THE REPAIR AREA USING THE LOCALIZED HEAT SOURCE:

149°C (300°F) FOR 32 HR MINIMUM
 THE 2219-T87 ALUMINUM SURFACE MUST NOT EXCEED 154°C (310°F)
 THE LINER TEMPERATURE MUST REMAIN ABOVE 144°C (290°F)

Figure 57. Sponge Pressure - Small Repair Procedure

These panels were cured at 52°C (125°F) for 16 hours under sponge pressure then post-cured at 149°C (300°F) for 32 hours without applied pressure. The tensile bond strength specimens taken from these panels were tested at cryogenic temperature and at elevated temperature [hot plate heating while holding 0.69 MN/m² (100 psi) constant load]. The results of these bond strength tests on double thickness adhesive are presented in Table 27. For comparison purposes, other panels using clean anodized aluminum plates were bonded to 3D foam using the sponge pressure method during cure but having only one adhesive layer. The results of the bond strength tests on single thickness adhesive are also presented in Table 28. Evaluation of the Epon 328/CL glass liner bond to 3D foam was also performed using the sponge pressure method in identical manner as described for the 211A/LZ adhesive. The liner-3D foam bond strength test results are presented in Table 29.

The tensile bond strength values associated with double adhesive layers of 211A/LZ and bonding pressure applied through compressed sponge polyurethane showed that no significant loss in strength will result from using this repair method in small areas of the tank insulation. The Mystik No. 5812 (Mystik Products Co.) tape used to hold the compressed sponge against the liner will slowly lift at temperatures over 55°C (130°F), however, and must be limited to a maximum size sponge pad of only 10 x 10 cm (4 x 4 inches).

The liner-to-3D foam bond line, when cured using only sponge pressure during the cure cycle, was unable to hold the constant load of 0.69 MN/m² (100 psi), for the 5 minute period required to heat the bond line to elevated temperature. This is indicated in Table 29 showing the temperature at which rupture occurred was only ~93°C (~200°F). The same low strength condition was experienced using 0.017 MN/m² (5 in. Hg) vacuum bag pressure reported in Table 13, Section 2.3.6, and is related to the unusually small fillets formed at the Z fiber contact points. The fillets at the fiber ends of the 3D foam used in small repair areas of the liner can be increased using a variety of techniques including the addition of Loctoweld 211A/LZ adhesive under the liner. The cost advantages of using sponge pressure pads in place of using vacuum bag pressure for small repairs is sufficient to offset the retention of this pressure application method.

Table 27
EFFECT OF SPONGE PRESSURE DURING CURE ON DOUBLE LAYER 211A/LZ (REPAIR)

Tensile Strength at -196°C (-320°F) MN/m ² (psi)	Temperature at Rupture Under Stress of 0.7 MN/m ² (100 psi) °C (°F)
1.20 (174)	185 (365)
1.19 (172)	188 (370)
1.39 (202)	181 (358)

- The second 211A/LZ adhesive layer was bonded to the cured first layer
- Rupture occurred at 2219-T87 surface — no separation between bond lines

Table 28
EFFECT OF SPONGE PRESSURE DURING CURE ON
SINGLE LAYER 211A/LZ (REPAIR)

Hold at Temperature Prior to Applying Pressure			Tensile Strength at -196°C (-320°F)		Temperature at Rupture Under Stress of 0.7 MN/m ² (100 psi)	
Temperature °C	(°F)	Time (hr)	MN/m ²	(psi)	°C	(°F)
25	(77)	1	>2.32 ^b	>(337)	184	(364)
			>1.20 ^b	>(174)	>188 ^b	>(370)
			1.73	(251)	173	(344)

^b = Specimen ruptured against tensile block or between tensile block and metal plate.

Table 29
EFFECT OF SPONGE PRESSURE DURING CURE ON
EPON 828/CL GLASS LINER (REPAIR)

Hold at Temperature Prior to Applying Pressure			Tensile Strength at -196°C (-320°F)		Temperature at Rupture Under Stress of 0.7 MN/m ² (100 psi)	
Temperature °C	(°F)	Time (hr)	MN/m ²	(psi)	°C	(°F)
25	(77)	1	1.57	(227)	93	(200)
			0.79	(115)	Ruptured while holding load and before heat could rise on bondline	
			0.87	(126)		

Repair of areas larger than can be covered with the sponge pad must be accomplished using vacuum bag pressure. In such cases, the vacuum bag must cover the repair and extend at least 30 cm (12 inches) over the edge of the repaired area per Figure 58 in order to offset the air leakage through the porous liner.

The syntactic foam gap filler mixture used between joints of 3D foam and described in Section 2.5.2, Figure 52, would be used to fill localized crushed areas, less than 2.5 cm (1.0 inch) diameter, where the liner is intact. Such areas must be considered as representing a debond of the liner and repaired by injection of gap filler. This formulation is repeated below and is compatible with the liner when subjected to cryogenic contraction loads.

- 70 parts Epon 828/CL resin
- 18 parts phenolic microballoons
- 12 parts glass milled fibers

2.6.1 Methods for Applying Heat to Cure Repair Areas of Insulation

Repair operations conducted during the tank insulation bonding operation would use the heating equipment normally provided in the bonding chamber

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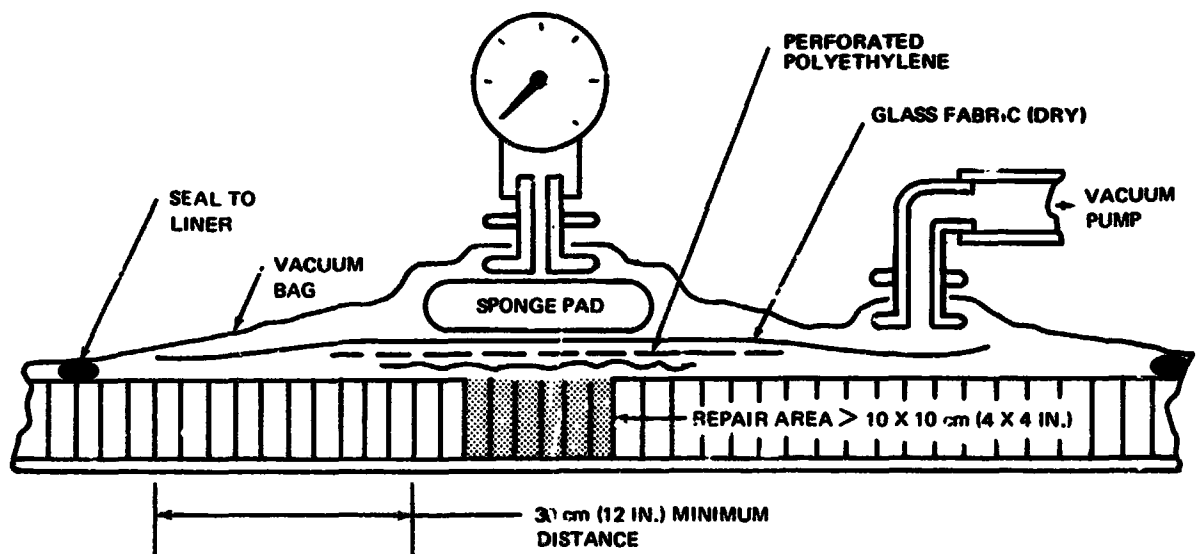


Figure 58. Vacuum Bag Pressure During Cure (Repair)

for curing the repaired areas. Field repairs would be more difficult to cure at 149°C (300°F) and maintain the temperature within ±6°C (±10°F) for 32 hours. A review of the heating methods that may be used to cure localized areas of insulation when conducting field repairs include the following heat sources:

- *1. Infra-Red Lamp Heater (Electrical)
(Incandescent light bulb for 52°C; 125°F)
- *2. Quartz Lamp Heater (Electrical)
- 3. Hot Air Blowers (Ducted from Source)
- 4. Hot Plate Direct Contact (Electrical)
- 5. Flexible Blanket Woven with Nichrome Wire (Electrical)
- 6. Hot Water Bladder (Ducted from Source)
(Only to 82°C; 180°F) or hot water
in plastic tubes mounted in a flexible
pad assembly
- 7. Steam Tubes Mounted as a pad assembly (Ducted from Source)

The primary factors involved with applying heat to localized areas of repair in the field are associated with safety. The use of electrical heating equipment inside the confines of the tank is hazardous to personnel safety. Pneumatically operated tools are commonly used inside the tank. Electrical safeguards are awkward to install and maintain and provide only partial protection against flash fires. In spite of these disadvantages, infra-red lamps and quartz heaters are currently used to apply heat to cure repair areas up to 77°C (170°F). With additional care and thermal shielding the temperature of 149°C (300°F) should be obtained with the same method.

The heating problem inside the tank to cure liner repairs is also compounded by the low thermal conductivity of the insulation. Local hot spots develop quickly and do not dissipate laterally. Temperature override thermocouples must be positioned at 7.6 cm (3 inch) intervals over the liner surface when infra-red or quartz lamps are used to apply heat.

*Asterisk indicates methods currently used to heat cure field repairs on 3D foam insulation up to 77°C (170°F).

The problems associated with heat lamps apply also to woven wire heating blankets with the additional disadvantage of obscuring the liner from visual observation.

The warm cure minimum temperature of 52°C (125°F) for 16 hours as recommended in Section 2.3.5 is only to allow personnel to operate inside the tank during warm cure. Localized repair operations may be heated directly to 149°C (300°F) if the repair is shielded from personnel exposure and if the pressure (vacuum bag) can be maintained on the repaired area until Shore Durometer hardness of over A100 is obtained on the adhesive. This would allow the heat to be applied only from the outside surface of the tank where the heat source would not be restricted by personnel safety to the degree required for internal operations.

The heating methods employing the use of external heat generators such as hot air blowers, hot water or steam offer the least hazard to personnel safety and the most desirable temperature control conditions relative to uniformity. The ducting may be awkward to handle inside the tank but if this heating method is used externally the ducting would present no more than conventional problems.

2.6.2 Non-Destructive Examination of Bond Lines 3D Foam-to-Tank Wall, 211A/LZ Adhesive

Debonds between the 3D foam adhesive and the aluminum tank wall are detectable using a pulse-echo ultrasonic unit consisting of the following elements:

Reflectoscope UM-700	Sperry Products
Transigate E550 Alarm System	Sperry Products
Transducers, 5.0 MHz--1.9 cm (3.4 in.) dia.	Sperry Products
L. S. Flat Contact	

The vibrating crystal or transducer is placed in intimate contact with the exterior, bare, metal surface using a glycerine solution coupling agent, and the returning energy converted to electrical signals is displayed on a reflectoscope. Operation of this instrument is illustrated in Figure 59.



Figure 59. Ultrasonic Examination of 3D Foam-to-Tank Wall Bond

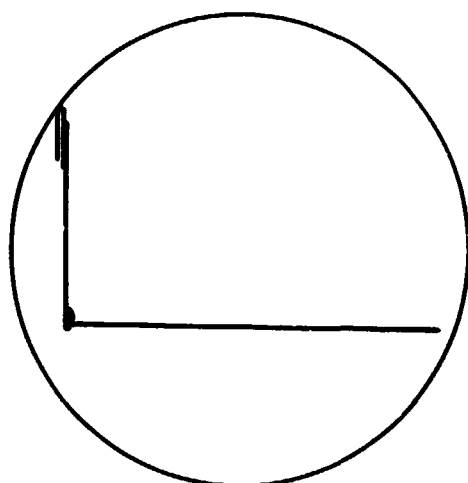
In order to determine the effectiveness of the pulse-echo ultrasonic inspection method using the insulation material construction developed in this program, a series of reference panels was fabricated. The debond conditions were prepared using two methods. The "porous" adhesive having 25 and 50 percent void areas were made by placing 0.31 cm (0.12 inch) squares of tape on the aluminum surface then spreading the adhesive over the entire panel. After a setting time of 17 hours at 25°C (77°F) the tape squares were peeled off and a slice of 3D foam pressured gently into the adhesive so as to make contact but not flow the adhesive into the bare spots left by tape removal.

Complete debonds of 1.3 cm (0.5 inch) and 2.5 cm (1.0 inch) diameter were fabricated by first punching the hole through the slice of 3D foam. The adhesive was spread over the 3D foam surface and bonded to the aluminum plate. The holes within the 3D foam were then filled with core plugs of 3D foam having a precured adhesive layer bonded over the bottom face. The precured adhesive on the core plug was forced against the metal plate but not bonded to the plate. A glass liner was bonded to the 3D foam on all panels to complete the composite construction.

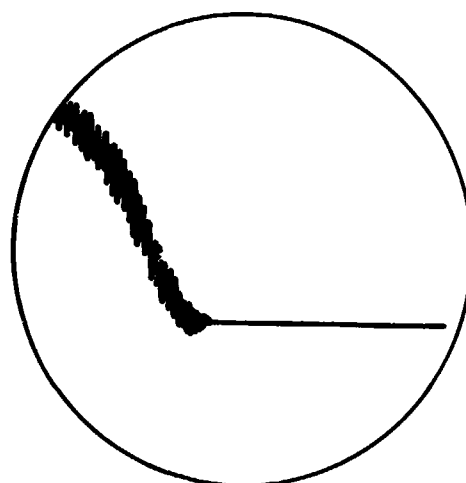
The aluminum plate thickness used for ultrasonic reference panels of this type should be exactly the same as the thickness of the tank wall being examined. However, the two thicknesses of aluminum used for evaluation of the method, 0.20 (0.08) and 0.64 cm (0.25 inches), demonstrated that pulse-echo ultrasonic equipment can discriminate between porous bond lines and completely debonded areas with Lefkowied 211A/LZ. The discrimination level was the same as that obtained when using Lefkowied 109/LM-52 adhesive applied to Saturn S-IVB LH₂ tanks and required very little change in frequency adjustments. The reflectoscope display representing porous adhesive bonds and complete debonds is illustrated in Figure 60.

2.6.3 Examination of Debonds Between Glass Liner and 3D Foam, Epon 828/CL

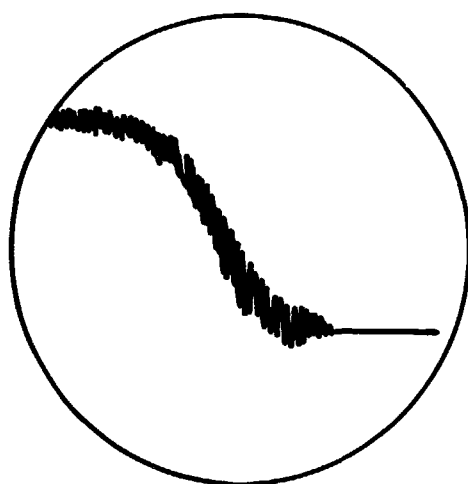
Debonds between the glass liner and the 3D foam of the Saturn SIVB LH₂ tank that are 1.3 cm (0.5 inch) diameter or larger in size are detectable using the sonic brush unit. The sonic brush is illustrated in Figure 61 and consists of a circular brass wire brush having a microphone suspended



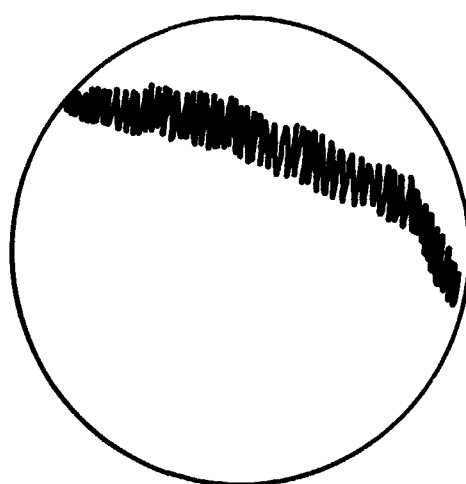
**STD NO. 1
GOOD BOND**



**STD NO. 2
25% DEBONDED
(POROUS ADHESIVE)**



**STD NO. 3 50% DEBONDED
(POROUS ADHESIVE)**



**STD NO. 4
COMPLETELY DEBONDED**

Figure 60. Ultrasonic Patterns from Reference Panels 211A/LZ Adhesive to Aluminum

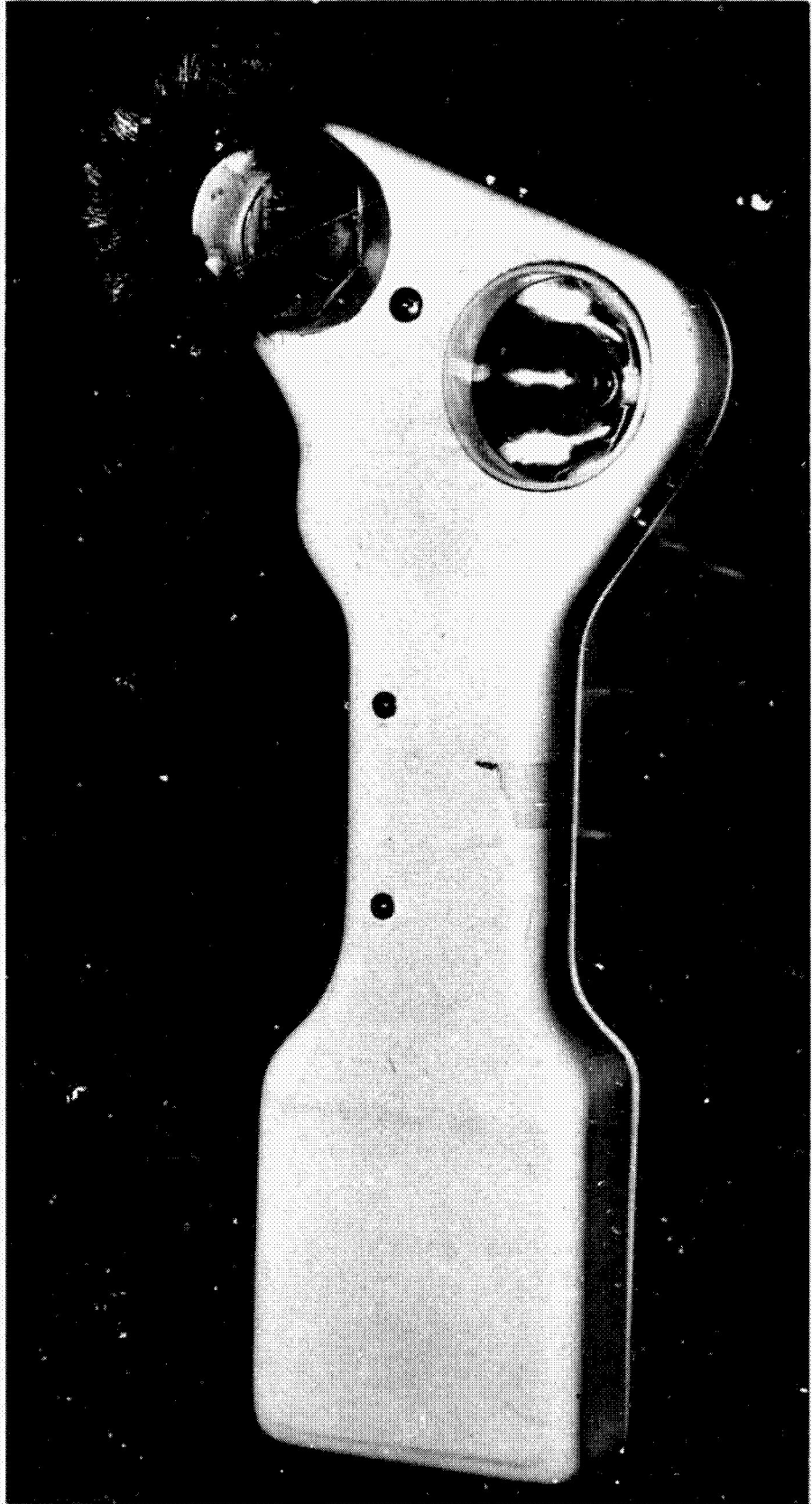


Figure 61. Sonic Brush

inside and just above the wire ends. This brush is moved laterally against the liner surface being examined and the microphone receives only the sound emitted from the wire ends. This signal is amplified and registered by the operator using a headset receiver.

The low frequency sound developed on a liner that is well bonded is easily distinguished from a liner that is completely debonded; however, the discrimination sensitivity must also be such as to provide identification of areas in which the liner is bonded only to the Z fibers and without bond to the foam between fiber contact points. This liner bond condition is not common but it does occur occasionally and will develop acceptable bond strength.

In order to determine the effectiveness of the sonic brush method for detection debonds using the 828/CL bonded liner developed in this program, a series of reference panels was fabricated. The foam was scraped away from the Z fibers to a depth of 0.30 cm (0.12 inches). Then the liner was bonded in place over the 3D foam to produce the most discriminating reference panel, having acceptable tensile bond strength, but where the liner was not bonded to the foam. Debonds of 1.3 cm (0.5 inch) and 2.5 cm (1.0 inch) diameter were produced in other fully cured sandwich panels by rupturing the fillet area by lightly tapping with a hammer then lifting the liner using an air nozzle.

The sound box created by the completely debonded areas was easily detected using the sonic brush detector. The hard smooth surface of the Epon 828/CL resin rendered distinction more difficult between the 1.3 cm (0.5 inch) diameter debond and the area having only Z fiber contact, and frequent cross examination of the reference panels was necessary in order to make positive identification.

An audio frequency discriminator providing an automated display with a recording unit could be used with the sonic brush but would sacrifice the portability of the unit and would unnecessarily increase the cost of tank liner examinations.

2.7 SURFACE PREPARATION (TASK 7)

The objective of this task was the preliminary evaluation and selection of corrosion-resistant, surface-coating systems for tank walls. The coating is to prevent corrosion of the tank wall surface between the time the tank sections are fabricated and the time that the insulation is installed. The requirement was to select a surface-preparation procedure that would provide the required corrosion resistance as well as adequate tank-wall bond mechanical properties consistent with other system requirements.

The baseline surface selected for adhesive bonding was the as-machined metal surface with a chromic-acid anodized coating. The preference of anodize coating was based on the following factors:

- Anodize produces a relative pinhole free corrosion resistant coating having a known high degree of reliability
- Production facilities exist for anodizing large panels
- The 2219-T87 aluminum is not subjected to elevated temperature bake cycles. Hot water rinse is only 88 °C (190 °F). Some primers require oven bake at 149 °C (300 °F).
- Anodize produces the lowest weight corrosion protection coating. Some primers will add as much as 0.61 kg/m² (0.013 lb/ft²) of tank surface.
- Primer coatings are highly sensitive to the adhesive type selected for subsequent bonding operations, and would have to be changed to accommodate other adhesives selected downstream of the vehicle design and production.

The test program described in detail in the appendix was conducted by MDAC independently on IRAD to establish confidence that 2219-T87 aluminum alloy for Shuttle applications could be anodized using the same manufacturing facility as was used to anodize 2014-T6 and other aluminum alloys used throughout MDAC operations. During this test program, the bondability of the anodize using Lefkowitz 211A/LZ adhesive was evaluated on each panel along with the corrosion resistance. The results of the tensile bond strength tests and corresponding corrosion resistance are summarized in Table 30 with a brief summary of the anodize processing variables.

Table 30
ANODIZE STUDY

Part of Program	Alloy and Surface	Anodize Date	Anodizing Time (min)	Seal Time (min)	Coating Weight gm/m ² (mg/ft ²)	Percent Hydration (PCH) (%)	Salt Spray Resistance (hr)	Average Tensile Strength at -196°C (-320°F) MN/m ² (psi)
1 Base Line Comparison	2219-T87 machined	1/18/72	30	0	1.91 (178)	--	--	--
				10	2.43 (226)	21.7	>1040	>1.82 (>263)
	2219-T87 chem milled	1/18/72	30	0	1.70 (158)	--	--	--
				10	2.07 (192)	17.5	>1040	>1.30 (>188)
2 Anodize Time Seal Time Study	2014-T6 chem milled	1/18/72	30	0	1.92 (178)	--	--	--
				10	2.44 (227)	21.7	>1540	>1.37 (>199) ^a
	2219-T87 machined	1/28/72	20	0	2.16 (201)	--	<42	--
				5	2.63 (244)	16.3	480	0.86 (125) ^a
				10	2.63 (244)	11.9	480	1.50 (217)
				20	2.67 (248)	13.7	480	>1.93 (>280)
		1/25/72	30	0	2.24 (208)	--	<60	1.42 (25)
				5	2.96 (275)	21.7	575	1.26 (183) ^a
				10	3.02 (281)	27.8	575	>1.81 (>262)
				20	2.96 (275)	26.8	575	>1.31 (>190) ^a

^a Average includes one or two values of less than 0.35 MN/m² (50 psi).

Table 30
ANODIZE STUDY (Continued)

Part of Program	Alloy and Surface	Anodize Date	Anodizing Time (min)	Seal Time (mi.)	Coating Weight gm/m ² (mg/ft ²)	Percent Hydration (PCH) (%)	Salt Spray Resistance (hr)	Average Tensile Strength at -196°C (-320°F) MN/m ² (psi)
2 Anodize Time Seal Time Study (Continued)	2219-T87 machined	2/3/72	45	0	2.83 (263)	--	<48	----
				5	3.72 (346)	24.6	>600	>1.49 (>216)
				10	3.87 (360)	27.4	>1100	>1.64 (>236)
				20	4.02 (373)	28.6	>1700	>1.82 (>263)
3 Production Run	2219-T87 machined	3/1/72	30	10	2.70 (252)	23.0	500	1.54 (223)
					2.91 (271)	24.8	336	1.26 (182)
		3/6/72			2.86 (266)	24.2	600	0.61 (89) ^a
		3/8/72			2.71 (252)	26.9	>600	1.38 (200)
		3/13/72			3.14 (292)	25.0	>672	1.26 (183)
		3/16/72			2.91 (271)	22.8	>672	1.40 (203)
		3/17/72			2.85 (265)	23.3	672	1.51 (219)
		3/18/72			2.90 (270)	23.1	--	----
		3/22/72			2.50 (241)	22.1	--	----
		3/23/72			2.94 (273)	23.8	528	----

^a Average includes one value of less than 0.35 MN/m² (50 psi).

No specific attempts were made to produce anodize coatings that were not bondable using 211A/LZ adhesive. The types of solution contamination or abnormal processing conditions that may result in anodized surfaces to which adhesive will not bond are indefinable and exploration in such abnormal areas would not constitute valid production conditions. As the results indicate, generally, no difficulty was experienced in achieving acceptable tensile bond strength with anodized 2219-T87 panels processed during the 3 month time encompassed by this particular test program during which the normal production variables were encountered and were measured.

The motivation for engaging in this exhaustive test program to evaluate production anodizing variables using 2219-T87 alloy stemmed from an isolated condition found with 2014-T6 alloy in which the anodize coating was not bondable to Leskoweld 109/LM-52 adhesive when tested at -197°C (-320°F). For this reason all tensile bond strength determinations were conducted at -197°C (-320°F).

2.8 TANKING TEST PLAN (TASK 8)

The objective of this task was to prepare a preliminary draft of a test plan for a subscale tank (of 2 to 4 m diameter). The test plan will form a sound basis for any subsequent subscale tank test programs, although such a program was beyond the scope of the Phase II effort.

The customary sequence, for development of a new cryogenic internal insulation or to qualify a change in configuration, is begun with laboratory coupon tests. MIL-STD-401-type coupon tests are required to screen candidate materials or configuration changes and to obtain material property/design data. The next level of testing has employed a 1 meter (3-ft) diameter dish-shaped plate in a large LH₂ cryostat. The cryostat and purge system are designed to accept catastrophic rupture of the insulation in the LH₂ without serious damage to the test facility. This test was utilized in Phase I to evaluate a Shuttle insulation panel under simulated tanking, pressurization, and reentry loadings. The cost of conducting the 1 meter dome test has been commensurate with the value of knowledge acquired on the integrated performance of liner, foam and bonds under LH₂.

Prior to approving the insulation system for flight tanks, a subscale tank, typically 2.4 meter (8 ft) diameter, is insulated using the manufacturing methods proposed for production tanks. This subscale tank is subjected to simulated environmental and stress conditions representing the most severe flight environments that can be predicted.

A primary need for a subscale tank test program is based on firm requirements to develop cost-effective manufacturing operations compatible with the adhesive and resin working properties. The subscale tank has proven to be a fully acceptable and low cost vehicle with which to accomplish this requirement. Manufacturing issues that must be evaluated and verified using a subscale (2.4 meter; 8 ft) tank prior to building full scale flight tanks include:

- A. The planned sequence of bonding contemplated for insulating flight tanks is also used for insulating the subscale tank, and is then analyzed for adaptability to flight tank insulation operations. The basic bonding sequence for a particular insulation composite must be firmly established, subscale, prior to insulating flight tanks in order to prevent a costly waste of man hours or a sacrifice of bond strength. Removal and replacement of a segment of poorly bonded production insulation has cost several times more man-hours than required for insulating the entire subscale tank and must be avoided by thorough evaluation of the bonding sequence using the subscale tank.
- B. The full extent of repair operations that may be required by a particular composite is not realized until completion of a subscale tank program. After testing the subscale tank, these repairs are validated as acceptable to be performed on flight tank insulation. Information on the type of repair required is not obtainable on small 1 meter (3 ft) dome insulation nor would a successful dome test necessarily validate the repair used for flight tanks.
- C. The handling abuse incurred during large scale installations, including walking on the "wet" adhesive coated 3D foam tile and the resin impregnated liner while installing the vacuum bags prior to curing operations, can only be evaluated by insulating a subscale tank. In contrast, it has always been possible to handle the 1 meter

dome insulation tenderly so as to minimize adverse side effects that would detract from a proper evaluation of the materials and the composite performance during cryogenic testing operations.

- D. Continuous mechanical mixing of adhesive and hardener is required for insulating subscale tanks. Resolution of problems using these mixing units relative to reliability must be resolved by subscale tank insulation operations. Dummy runs using the mixing machines without the critical time element associated with intermittent use during bonding operations have limited value in judging their capability. The bonding operation on 1 meter (3 ft) dome insulation requires only 0.9 kg (2 lb) of adhesive over a one-hr period and can be easily handled manually. In general, knowledge of the influence of manufacturing facility and equipment variables on the quality and characteristics of the installation is necessary prior to full scale fabrication. The differences between laboratory and manufacturing facility hardware can be significant.
- E. Fabrication of a subscale tank is necessary in order to evaluate special tile and joint designs and fabrication procedures for protrusions, weld joints, close-outs, and attachments which cannot be scaled down to a (1 m) dome.

As stated before, a subscale tank test would provide verification of the structural integrity of the insulation and data on the thermal performance of a production-type installation. It would also provide verification of installation procedures and training for installation personnel.

The following is a plan for constructing and testing a subscale tank based on a plan used in developing the S-IV insulation.⁹

The tank would be constructed with an interior surface similar to that for a full scale vehicle, including isogrid if that is to be used. The subscale tank would be made by machining the cylindrical section using modified Thor vehicle tooling welding, Thor oxygen tank domes to the ends of the cylinder. Necessary provisions would be made for a manhole, instrumentation, plumbing, etc. Thor transportation facilities and a Thor test complex would be used. A possible test tank configuration is shown in Figure 62. General

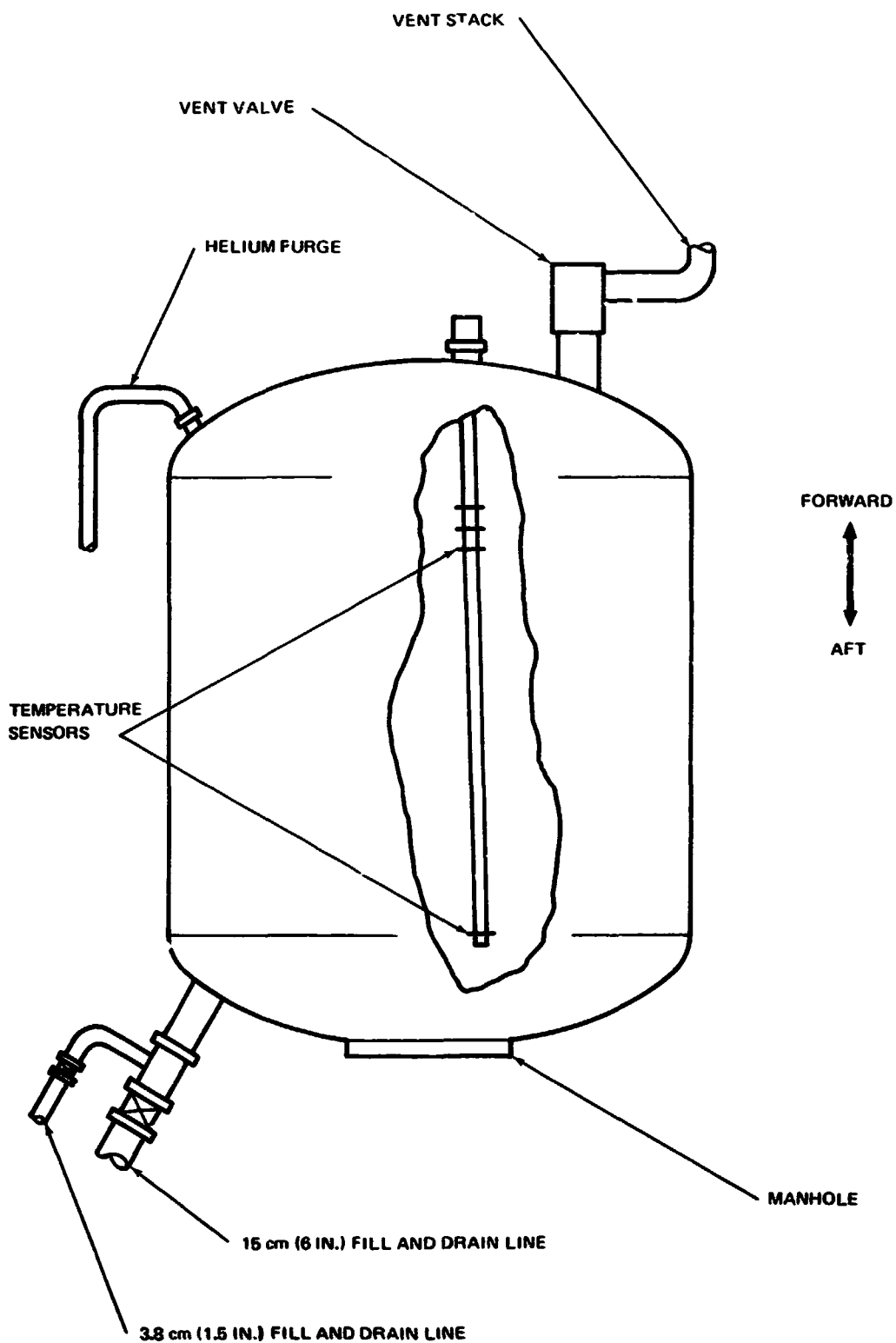


Figure 62. Test Tank – 2.4 m (8 FT)

dimensions and possible temperature sensor locations (T/S) are indicated in Figure 63. Sensors would be located on a probe near the center of the tank, as well as on the liner surface and the outside surfaces of the tank. The domes would have a radius of about 2.03 m (80 in.).

Shuttle prototype foam insulation, BX-251A-3D, would be bonded to the interior tank surface with Lefkowlad 211A/LZ adhesive. The inner liner (adjacent to the liquid) surface of the 3D foam would be covered with one layer of 116 glass cloth impregnated and bonded to the 3D foam using Epon 828/CL epoxy resin. The complete surface would then be given one rub coat of Epon 828/CL.

The completed tank including insulation would be attached to a Thor launcher in the horizontal position. The tank would be mounted to an empty Thor engine section illustrated in Figure 64 to allow for attachment of the test tank to the launcher which would have the capability of erecting and lowering the tank. The tank would have complete plumbing for filling and draining of the LH₂.

Figure 62 shows the locations of the temperature probe (used to record the liquid level inside the tank) and the tank differential pressure transducers.

The frost thickness will be periodically observed by viewing through a transit a depth indicator attached to the tank surface. Atmospheric conditions will be obtained periodically from the nearest weather station (selected in advance).

Two methods may be utilized to calculate boiloff. The first uses the change in the pressure differential (ΔP) recorded during boiloff which reflects a change in the hydrostatic head. The second method is based on the time consumed by the liquid traveling from one level to another. The temperature sensors would be used to indicate the presence of the liquid. From previous experience there is a very good correlation between the two methods in determining the liquid level change with time.

The heat transfer would be evaluated using the temperature difference from the wall to the liquid and the measured boiloff rates. This method would account for changes in environmental conditions.

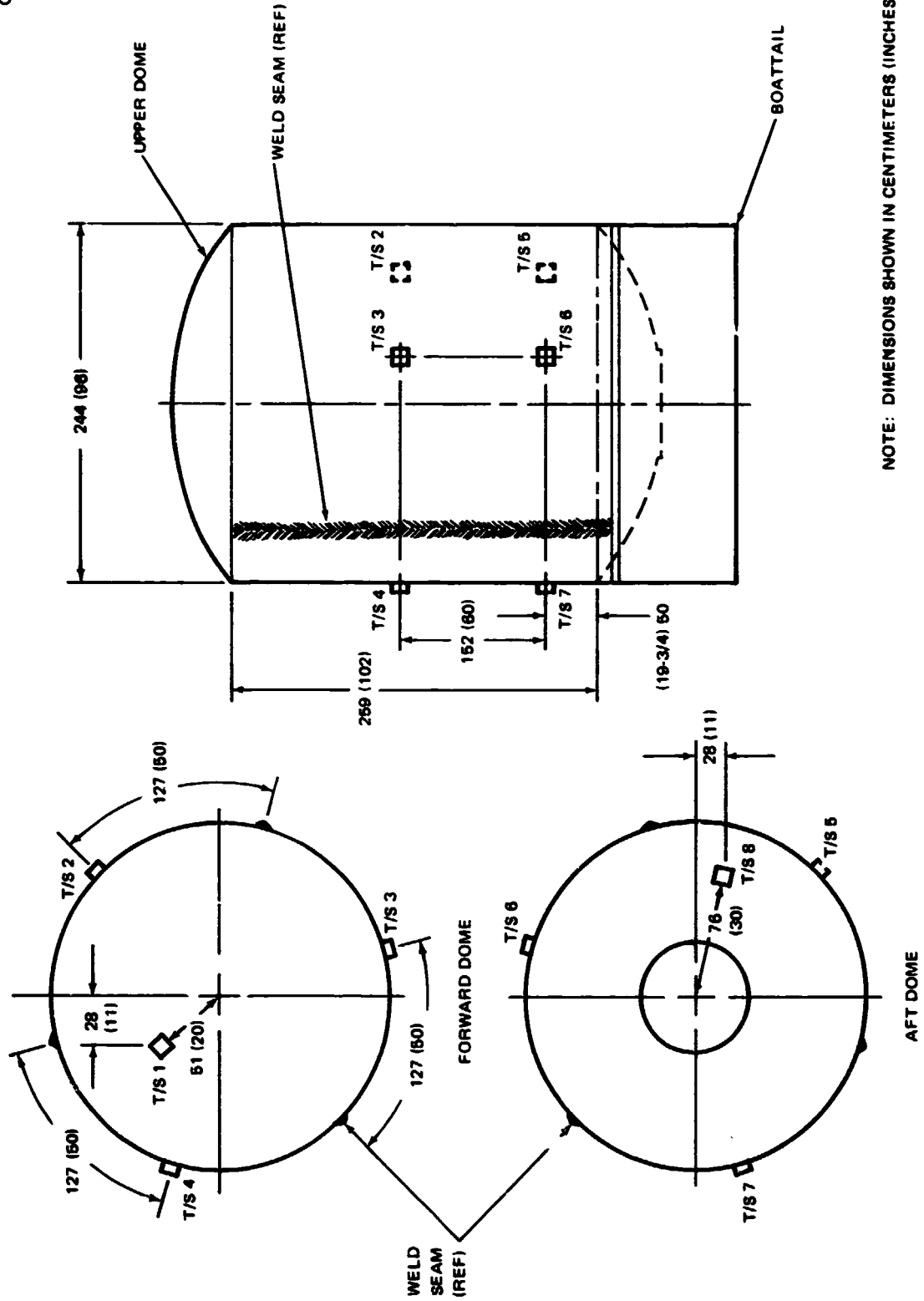


Figure 63. Temperature Sensor Location - 2.4 m (8 Ft) Scale Tank

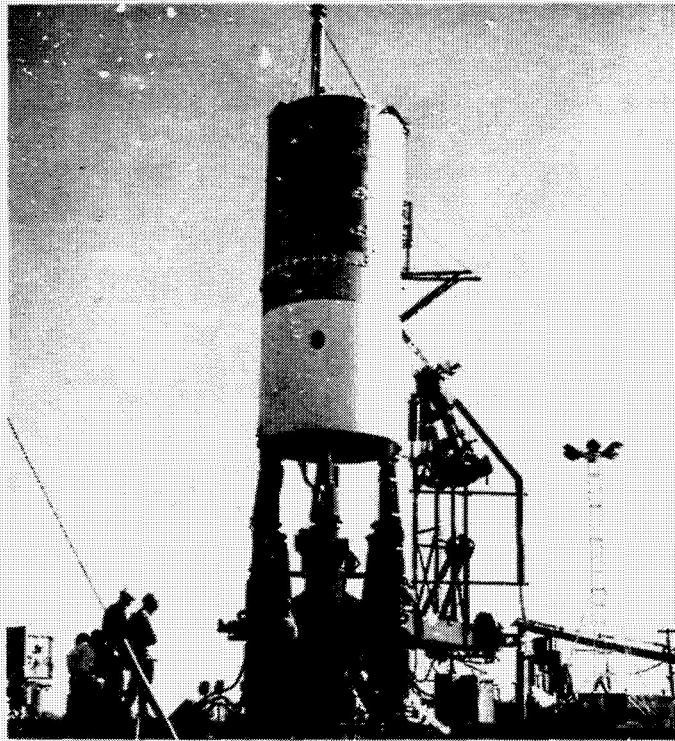


Figure 64. Subscale Tank Test

It is expected that the thickness and density distribution of the frost may cause considerable difficulty in evaluating the transient variation in wall temperature. During the S-IV subscale tanking tests it was noted that in some of the tests virtually no frost formed on the tank side walls while during others the frost was up to 0.64 cm (0.25 in.) thick. This frost had the effect of lowering the wall temperature by as much as 28°C (60°F) to 56°C (100°F). The frost distribution on the top and bottom domes would probably be different than for the tank sides.

The heatup and elevated temperature cycles should approximate those expected with a flight vehicle during normal or abort reentries. The temperature distribution around the circumference and along the length of the tank might be varied to simulate heating on the upper and lower surfaces of the vehicle in open areas and near attachments. The heating system should be capable of producing the required heating rates and temperature profiles.

Thermocouple data should be used to determine tank wall temperatures, liner temperatures and the elevated temperature thermal conductivity of the

3D foam. The magnitudes of stresses induced in the composite above and below the LH₂ level might be considered using analytical procedures as well as strain gauges or other strain measuring techniques.

A typical test procedure would consist of the following steps:

1. Fill the tank with LH₂ at a predetermined rate to a level of 2.6 m (8.5 ft).
2. Maintain the liquid level until thermal equilibrium is attained or approximately 10 hours.
3. Increase pressure using helium gas to develop shuttle tank wall stress or approximately 0.28 MN/m^2 (40 psi). Hold pressure for 60 seconds.
4. Reduce pressure to ambient.
5. Allow the LH₂ to boil off. Record pressure differential, tank wall temperature and liquid level simultaneously until the liquid level has decreased to 1.2 m (4.0 ft).
6. Drain the tank.
7. Heat the tank wall outer surfaces to simulate shuttle aerodynamic heating profile or approximately 177 °C (350 °F).
8. Allow the tank to cool to ambient.

Tests or Observations to be Performed

As Fabricated

1. Tensile bond strength at -196 °C (-320 °F) on flat panels bonded simultaneously with tank insulation. Liner and tank wall bonds.
2. Tensile plug tests at 25 °C (77 °F) on flat panels. Liner and tank wall bonds.
3. Tensile plug tests inside the tank. Liner and tank wall bonds at 25 °C (77 °F).
4. Ultrasonic examination of tank wall bond.
5. Sonic brush examination of liner bond.

After First LH₂ Pressurization and Heating Test

1. Repeat tensile plug tests inside the tank at 25 °C (77 °F) and compare results with flat panel plug tests.

2. Ultrasonic examination of tank wall bond.
3. Sonic brush examination of liner bond.

Repeat these tests after the second, fourth, sixth, thirteenth and twenty-fifth test cycles.

The results of these plug tests will be analyzed to establish the rate at which bond strength degradation occurred during 25 test cycles and extrapolated to estimate bond strength degradation after 100 test cycles. This information, along with ultrasonic and sonic brush examinations, will be used to judge the advisability of continuing the LH₂ cycle test program beyond the initial 25 cycles.

It is anticipated that sufficient data would be available from this subscale tank test program to predict the performance of a full scale shuttle tank insulation using ultrasonic examination from the outside surface and periodic examination of the liner inside the tank. Plug testing and subsequent repair operations inside the flight vehicles would be performed only in cases of exposure to abnormal flight stresses or abnormal temperature conditions.

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Section 3
SPECIFICATION REQUIREMENTS

The concept developed and characterized under Phase I is to be described by material and process specifications at contract completion. Phase II is designed to develop the data for material and process requirements so that the insulation system may be practically and efficiently installed in full-scale tanks. Detailed specifications are to be submitted at the completion of Phase III under separate cover.

3.1 FOAM

The foam materials and process specifications will cover the following items:

- A. Foam components and storage
- B. Foam mixing
- C. Reinforcing fibers
- D. Fiber impregnating material
- E. Fiber array
- F. Material characterization and quality control testing.

3.2 BONDING

The bonding materials and process specifications will cover the following items.

- A. Anodizing and tank wall surface preparation
- B. Tank wall adhesive components and storage
- C. Adhesive mixing
- D. Adhesive application
- E. Liner materials
- F. Liner impregnating resin components and storage
- G. Impregnating resin mixing
- H. Installation of 3-D foam
- I. Impregnation and installation of the liner
- J. Vacuum bagging and insulation system cure cycle
- K. Characterization and quality control testing

3.3 INSULATION REPAIR

The insulation repair specification will provide for in-plant and field repairs to weak or damaged insulation. Repairs requiring only the replacement of the liner will be considered as well as repairs requiring the replacement of the insulation composite in small areas.

Section 4

SUMMARY AND CONCLUSIONS

The following is a summary of the significant accomplishments, observations, and conclusions achieved during the reporting period.

3D Foam

- BX-251A-3D foam was found to offer superior performance to BX-249N for Shuttle 3D internal insulation applications. BX-251A-3D was substituted for BX-249N-3D as the baseline insulation.
- BX-251A-3D was shown to meet Shuttle insulation requirements at a density of only 43 kg/m³ (2.7 PCF).
- Quality control methods were established for production manufacturing operations with BX-251A-3D.

Lefkowitz 211A/LZ Adhesive and Epon 828/CL Glass Liner

- Catalyzed working life at various "cold" temperatures using L211A/LZ and 828/CL bonded to 3D foam was shown to allow Shuttle size LH₂ tanks to be insulated using only one or two bonding sequences as compared to the 25 sequences used for Saturn S-IVB insulation. This will result in a significant cost reduction in bonding large tanks.
- Production-quality adhesive mixing parameters and weight were identified. Production-quality mechanical mixing equipment for L211A/LZ was procured and verified as capable of production bonding operations.
- Production-quality curing and bagging processes were established along with their tolerances for each bond line (tank wall and liner). Simultaneous bonding operations of wall and liner were shown to be compatible. A final cure, without vacuum bag pressure, is required at 149°C (300°F) to obtain acceptable bond strength at 177°C (350°F).
- Quality control methods and criteria were established for production bonding operations.

Repair Operations

- The strength of simulated repair operations was demonstrated and recommendations made for field repair heating.

3D Foam Joints

- Structural and thermal analyses and tests demonstrated the integrity of the liner bonded over nominal unfilled and filled, rigid joints in 3D foam. Both butt joints and shiplap joints can be produced and are thermally and structurally adequate.

Vibration and Acoustic Response

- Vibration and acoustic environments for the Shuttle internal insulation were defined and stresses predicted. A fatigue test program, indicated as valuable in the ultimate qualification of the Shuttle, was defined.

Anodize Coating of 2219-T87 Aluminum Alloy Tank Wall

- Corrosion resistance and bondability to L211A/LZ adhesive of chromic acid anodized 2219-T87 were demonstrated. Isolated anomalies remain to be evaluated.
- Production anodize process variable interactions were evaluated. It was shown that reasonable production tolerances in processing variables can be accepted.

Section 5

REFERENCES

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Appendix SURFACE PREPARATION

This appendix is divided into two sections. The first deals with the selection of a surface preparation and the second with an in-house IRAD program to characterize MDAC chromic acid anodized 2219-T87 aluminum.

1.0 SELECTION OF BASELINE SURFACE PREPARATION

The baseline surface selected as a result of Phase II, Task 2.7 was as-machined 2219-T87 with a chromic acid anodized coating. The decision was reached in conjunction with the NASA Contracting Officer Representative (COR).

Surface Preparation Selection

The approach used was to screen various tank surface preparation methods using the tensile bond test and L211A/LZ adhesive, and to select—mutually with NASA/MSFC—a method compatible with good engineering and manufacturing practices.

The surface preparation task was carried out using the following steps:

- A. Fabricate BX-249N-3D-S foam panels 2.5 by 30.5 by 30.5 cm (1.0 x 12 x 12 in.)**
- B. Machine 2219-T87 aluminum plates 0.20 by 30.5 by 30.5 cm (0.080 by 12 by 12 in.)**
- C. Leave some of the panels in the as-machined condition. Chemically mill approximately 50 microns (2 mils) of material from each side of some of the panels. Grit blast some of the panels.**
- D. Solvent wipe or vapor degrease the panels and then anodize or spray coat with primer. Cure the primed panels as required.**
- E. Determine coating weights and salt spray lives for the coated and anodized panels.**
- F. Fabricate tensile bond specimens using L211A/LZ adhesive and standard procedures.**

- G. Tensile test the specimens in liquid nitrogen (I-N_2) and at elevated temperature.
- H. Select the baseline surface preparation procedure.
- I. Describe the preliminary surface preparation procedure.

The salt spray resistance data are summarized in Table A1. The combinations of coatings and metal surface conditions used for tensile testing are indicated in Table A2. The M602 and L211A/LZ (acetone) panels cited in the table were produced using nominal application procedures that were not optimized. The anodized panels exhibited coating weights between 2.0×10^{-3} and $4.0 \times 10^{-3} \text{ kg/m}^2$ (0.2 and 0.4 g/ft²) whereas the primers exhibited coating weights between 20.0×10^{-3} and $75.0 \times 10^{-3} \text{ kg/m}^2$ (2.0 and 7.0 g/ft²). The range in the primer coating weights may have been substantially reduced by optimized application procedures.

The elevated temperature and cryogenic tensile tests data are contained in Tables A3 and A4. Table A5 contains data comparing the tensile strength of 2014-T6 and 2219-T87 aluminum with L109/LM52 and L211A/LZ adhesives at -196 °C (-300 °F). L109/LM52 was used for bonding the SIVB insulation to the chemically milled anodized 2014-T6 aluminum tank wall. As machined anodized 2219-T87 was proposed as the material for the Shuttle LH₂ tankage and L211A/LZ was selected as the adhesive for bonding the internal insulation.

Based on the above data the as-machined, anodized surface was selected as the baseline surface preparation.

Following is the surface preparation procedure:

- A. Vapor-degrease or solvent wipe the as-machined metal surface as required.
- B. Chromic-acid anodize the surface.
- C. Immediately prior to installation of the insulation system, reclean the tank surface.

Table A1
SALT SPRAY RESISTANCE OF 2219-T87

<u>Surface Preparation</u>		Salt Spray Resistance (hours)
<u>Coating</u>	<u>Surface</u>	
MDAC Anodized (Std. Process) (Santa Monica)	As machined Chem. milled	500 500
DAC Anodized (Long Beach)	As machined	92
FR primer	As machined Chem. milled	600 504
M602 ^a	As machined Chem. milled	500 170
L211A/LZ ^a (acetone)	As machined	72

^aSystem not optimized

Table A2
COATING AND METAL SURFACE CONDITIONS
EVALUATED USING TENSILE BOND TESTS

Coating	Metal Surface		
	As machined	Chem. Milled	Grit blasted
None	X	X	X
MDAC Anodized (Std Process)	X	X	
FR Primer	X	X	
M602 Primer	X	X	
L211A/LZ (acetone)	X		
34127 Primer	X		

Table A3
TENSILE BOND STRENGTH ON AS-MACHINED
2219-T87 ALUMINUM SURFACES

Surface Preparation		Tensile Strength -196° C (-320° F)		Temperature at Rupture Under Stress of 0.7 MN/m ² (100 psi)	
Metal Surface	Coating	MN/m ² (psi)		° C	(° F)
As machined	None	2.11	(305)	160	(320)
		2.07	(300)	166	(330)
		1.96	(284)	172	(342)
				172	(342)
				152	(307)
				103	(218)
				146	(295)
		Average	2.04 (296)	Average	153 (308)
As machined	Anodized, MDAC (Santa Monica)	2.00	(290)	168	(335)
		2.21	(320)	179	(355)
		1.86	(270)	151	(304)
				152	(306)
		Average	2.02 (293)	Average	163 (325)
As machined + British etch	M602 primer	3.35	(486)	174	(345)
		2.10	(305)	126	(258)
		2.59	(375)	164	(328)
		Average	2.68 (389)	Average	154 (310)
As machined	FR primer MDAC	2.20	(318)	138	(280)
		1.81	(263)	132	(270)
		2.04	(296)	124	(256)
				108	(227)
		Average	2.02 (293)	Average	126 (258)
As machined	L211A/LZ (acetone primer)	2.04	(295)	135	(275)
		2.58	(374)	134	(273)
		1.71	(248)	131	(355)
		Average	2.11 (306)	Average	133 (272)
As machined	34127 primer (515-X301)	1.83 ^a	(265)	171	(340)
		1.90 ^a	(275)	172	(342)
		1.95 ^a	(282)	179	(355)
		1.90 ^a	(275)		
		Average	>1.90 (275)	Average	174 (346)

^aIndicates specimen failed primarily against grit-blasted tensile block or between tensile block and metal plate.

Table .
TENSILE BOND STRENGTH FOR ROUGHENED
2219-T87 ALUMINUM SURFACES

Surface Preparation		Tensile Strength -196°C (-320°F) MN/m ² (psi)		Temperature At Rupture Under Stress of 0.7 MN/m ² (100 psi)	
Metal Surface	Coating			°C	(°F)
Grit blasted	None	1.87 ^a	(271)	171	(340)
		1.52 ^a	(220)	182	(360)
				179	(355)
				182	(360)
				Average 179	(354)
Chemically milled	None	2.15	(311)	182	(360)
		1.74	(252)	182	(360)
				177	(350)
				166	(330)
				170	(338)
				168	(335)
				171	(340)
				152	(305)
Chemically milled	Anodize, MDAC (Santa Monica)	2.66	(385)	177	(350)
		2.59	(375)	185	(365)
				168	(335)
				160	(320)
				Average 172	(342)
Chemically milled	M602 primer	1.88	(272)	182	(360)
		1.74 ^a	(252)	177	(350)
				179	(355)
Chemically milled	FR primer, MDAC			Average 179	(355)
		1.61 ^a	(233)	163	(325)
		1.64	(238)	166	(330)
				166	(330)
				152	(305)
				Average 161	(322)

^aIndicates specimen failed primarily against grit-blasted tensile block or between tensile block and metal plate.

Table A5
BONDING COMPARISON AT -196 °C (-320 °F)

Aluminum Alloy	Adhesive	Surface Preparation	Tensile Strength at -196 °C (-320 °F)	
			MN/m ²	(psi)
2014-T6	L109/LM52	Chemically milled and anodized	1.81 ^a	(263)
			1.38	(200)
			1.21	(175)
	L211A/LZ	Chemically milled and anodized	1.69 ^a	(245)
			2.40	(348)
			1.90	(275)
2219-T87	L109/LM52	As machined and anodized	1.52 ^a	(220)
			1.59	(230)
			1.61	(233)
	L211A/LZ	As machined and anodized	2.42 ^a	(350)
			1.85	(268)
			1.67	(242)

^a Specimen failed primarily against grit-blasted tensile block or between tensile block and the metal plate

2.0 STUDY OF ANODIZE PROCESS VARIABLES

Aluminum alloy 2219 nominally contains 5.8 to 6.8 percent copper. This exceeds the 5.0 percent nominal copper limitations of MIL-A-8625C (Anodic Coating, For Aluminum And Aluminum Alloys) for Type I coatings. A Type I coating is, by definition, a chromic-acid-produced anodic coating.

2.1 BASELINE COMPARISON

In order to help qualify 2219-T87 to the corrosion requirements of the Mil Specification an independent in-house IRAD program was conducted at MDAC. The objective of the program was to evaluate the effects of several anodizing processing variables on the tensile bond strength, salt spray resistance, anodic coating weight, and percent hydration of anodized 2219-T87. The program was carried out in parallel with Phase II.

The tensile bond data resulting from the program are summarized in Section 2.7.

The anodizing part of the program was divided into three parts:

Part 1 – Baseline comparison of anodizing characteristics of 2219-T87 and 2014-T6 aluminum alloys having chemically milled or as-machined surfaces.

Part 2 – Study of effect of anodizing time and seal time on the anodic coating on machined 2219-T87 aluminum.

Part 3 – Study of typical variations in anodize quality resulting from normal production anodizing operations. Examine effect of stripping the anodize from a panel and then reanodizing.

Alloy 2014-T6 was used as a control since a good history of this alloy exists and any anomaly in chemical processing could be quickly detected. The 2014-T6 panels were initially about 0.23 cm (0.090 in.) thick and were chemically milled to about 0.20 cm (0.080 in.) thickness. The 2219-T87 as-machined panels were initially nominally 0.20 cm (0.080 in.) thick; in chem-milling about 0.005 cm (0.002 in.) was removed from each side.

The baseline process sequence was as follows:

1. Vapor degrease panels
2. Alkaline clean, rinse, triacid etch and again rinse.
3. Anodize for 30 min. at 40 volts with a 5 min. voltage rise time.
4. Rinse and then water seal for 10 min.

Coating weight determinations and 5% salt spray exposure tests were conducted per methods specified in MIL-A-8625. A chemical history of the processing solutions was maintained.

The anodizing and sealing were done in production tanks. The coating weight was determined using ASTM B 137 and the percent hydration (PCH) computed using the following equation:

$$P = \frac{\Delta w}{w + \Delta w} \times 100$$

where

P = percent hydration (PCH)

w = unsealed coating weight

w = coating weight gain due to sealing

The percent hydration (PCH) was determined by two methods:

Method A requires only one specimen. Coating weights and seal water pick-up are determined by weighing the anodized unsealed specimen, and reweighing after the sealing operation. The sealed panel is then stripped of its coating and the bare specimen is weighed a third time. Coating weights and water pick-up are determined by weight difference and PCH calculated.

Method B was developed primarily for production process control and requires one unsealed, and one sealed specimen for obtaining the coating weight and coating weight plus seal water pick-up. The PCH was calculated by assuming that the unsealed coating weight of both specimens was the same.

The results of Part 1, the baseline comparison, are summarized in Tables A6, A7, A8 and A9.

The data in Table A6 indicate that the unsealed and 10 min. sealed coating weights and PCH's are lower for chem. milled 2219-T87 than for chem. milled 2014-T6. The salt spray resistances indicated in Table A7 are well above the MIL Spec. 336 hour requirement. The tensile bond strength values indicated in Table A8 are comparable to the 1.4 MN/m² (200 psi) values previously obtained at -196°C (-320°F).

Table A9 shows the unsealed coating weights of specimens with respect to rack location during anodizing. The specimen positions are numbered from top to bottom. The variations due to position do not appear to be significant.

2.2 ANODIZE TIME – SEAL TIME COMPARISON

The anodize time-seal time comparison, Part 2, was performed in order to determine the effects of off nominal variations in anodizing and seal times on specimen coating weight, PCH, salt spray resistance and tensile bond strength. The results are summarized in Tables A10 and A11 and Figures A1, A2, A3 and A4.

Table A6

PERCENT HYDRATION (PCH) OF 30 MINUTE ANODIZED ALUMINUM
ALLOYS USED FOR "BASE LINE" TESTS PROCESSED 1/18/72

Rack No.	Number of Specimens Used	PCH Method	Unsealed Coat Wt. gm/m ² (mg/ft ²)	Seal Time Min.	Sealed Coat Wt. gm/m ² (mg/ft ²)	Percent Hydration (PCH)	Avg. PCH
2219-T87, Machined Surface							
1	2	B	1.89 (176)	10	2.56(238)	26.0	
2	2	B	1.89 (176)	10	2.44(227)	22.3	21.1
4	2	B	1.95 (181)	10	2.37(220)	17.6	
5	2	B	1.92 (178)	10	2.36(219)	18.6	
2219-T87, Chem-Milled Surface							
1	2	B	1.79 (166)	10	2.10(195)	15.0	
3	2	B	1.61 (150)	10	1.95(181)	17.1	17.5
5	2	B	1.70 (158)	10	2.13(198)	20.3	
2014-T6 Chem-Milled							
1	2	B	1.96 (182)	10	2.55(237)	23.3	
2	2	B	1.99 (185)	10	2.55(237)	22.0	
3	2	B	1.68 (156)	10	2.12(197)	20.7	21.7
4	2	B	1.99 (185)	10	2.52(234)	21.0	
5	2	B	1.97 (183)	10	2.50(232)	21.3	

Table A7
FIVE PERCENT SALT SPRAY RESISTANCE RESULTS: 30-MINUTE ANODIZE
"BASE LINE" TESTS FOR 2219-T87 and 2014-T6

Alloy and Surface Finish	Number of Specimens Used	Salt Spray Exposure Failure Time (Hrs)		Unsealed Coat Wt. gm/m ² (mg/ft ²)	10 Min. Seal Coat Wt. gm/m ² (mg/ft ²)	Percent Hydration (PCH)*
		Minimum	Maximum			
2219-T87 Machined	7	1040	1540	1.92(178)	2.43(226)	21.1
2219-T87 Chem-Milled	7	1040	1540	1.70(158)	2.06(192)	17.5
2014-T6 Chem-Milled	2	1540	1540**	1.92(178)	2.44(227)	21.7

*PCH determined by Method B

**Removed from test at listed test time in good condition

Table A8

-196°C (-320°F) TENSILE STRENGTH: PART 1 – BASELINE

Anodized Alloy	Surface Condition		
	As Received	As Machined	Chem. Milled
2219-T87		>405	20
		105	322
		278	>222
2014-T6	70		107
	>162		187
	>300		>302
> Specimen failed at other than anodize surface			

Chromic acid was added to the anodizing tanks between the time that the Part 1 specimens were anodized and the time that Part 2 anodizing was initiated, as a result the coating weights for Part 2 are considerably higher than for Part 1. The salt spray resistance, for 30 minute anodized 10 min. sealed 2219-T87 in Part 2 was considerably less than for the same material processed in Part 1, however the tensile bond strengths were comparable.

The salt spray resistance apparently increases with anodizing time for constant seal times (Table A10). For 20 and 30 minute anodizing times the data indicate that a 5 minute seal produces near maximum salt spray resistance, however for a 45 minute anodizing time salt spray resistance continues to increase with seal time. A number of specimens did not fail against the anodized surface. As a result the recorded average values are minimums. Although tensile data would indicate average values above 0.69 MN/m^2 (100 psi) individual specimens failed at values below 0.69 MN/m^2 (100 psi). The causes of these failures are currently the subject of continued investigation.

2.3 PRODUCTION RUNS

In order to determine typical production anodize reproducibility panels were anodized over a period of weeks. The specimens were processed by production personnel under production conditions. The effects of rework by

Table A9
UNSEALED COATING WEIGHTS, 30 MIN. ANODIZED ALUMINUM
ALLOYS, PROCESSED 1/18/72 ("BASELINE" TEST)
EFFECT OF RACK POSITION ON COATING WEIGHT

Rack Position	2219-T87, Machined Surface	2014-T6 Chem-Milled Surface
	Coat Wt. gm/m ² (mg/ft ²)	Coat Wt. gm/m ² (mg/ft ²)
1	1.81 (168)	1.93 (179)
2	1.85 (172)	1.99 (185)
3	1.83 (170)	2.02 (188)
4	1.81 (168)	2.01 (187)
5	1.83 (170)	2.02 (188)
6	1.85 (172)	1.98 (184)
7	1.87 (174)	2.04 (189)
8	1.83 (170)	2.01 (187)
9	1.82 (169)	1.89 (176)
10	1.81 (168)	2.00 (186)
11	1.78 (165)	2.11 (196)
12	1.77 (164)	2.00 (186)
13	1.75 (163)	2.00 (186)
14	1.78 (165)	2.02 (188)
15	1.75 (163)	2.08 (193)
16	1.77 (164)	2.03 (189)
Avg. Coating Weight 1.81 gm/m ² (168 mg/ft ²)		2.01 gm/m ² (187 mg/ft ²)
Coating Weight Range 1.75-1.87 gm/m ² (163-174 mg/ft ²)		1.89-2.11 gm/m ² (176-196 mg/ft ²)

Table A10

FIVE PERCENT SALT SPRAY RESISTANCE TEST RESULTS: 20, 30, AND 45 MINUTE
ANODIZED 2219-T87, MACHINED SURFACE

Anodize Time, Min.	Seal Time, Min.	Number of Specimens	Unsealed Coat Wt. gm/m ² (mg/ft ²)	Sealed Coat Wt. gm/m ² (mg/ft ²)	Percent Hydration (PCH)*	Salt Spray Exposure Failure Time, Hrs.	
						Minimum	Maximum
20	0	2	2.16(201)	---	---	24	42
20	5	2	2.20(204)	2.62(244)	16.3	480	480
20	10	2	2.32(215)	2.62(244)	11.9	480	480
20	20	2	2.30(214)	2.67(248)	13.7	480	480
30	0	4	2.24(208)	---	---	32	60
30	5	2	2.32(216)	2.96(275)	21.7	575	575
30	10	2	2.19(203)	3.02(281)	27.8	575	575
30	20	2	2.16(201)	2.96(275)	26.8	575	575
45	0	2	2.83(263)	---	---	24	48
45	5	2	2.81(261)	3.72(346)	24.6	600	1700
45	10	2	2.82(262)	3.87(360)	27.4	1100	1776**
45	20	2	2.86(266)	4.02(373)	28.6	1700	1776**

*PCH determined by Method B

**Still in test as of 4/21/72.

Table A11
-196°C (-320°F) TENSILE STRENGTH:
PART 2—ANODIZE TIME-SEAL TIME STUDY^a

Anodize Time (min)	Seal Time (min)			
	0	5	10	20
20		95		>265
		25		>260
		20	238	>315
		301	287	
		184	125	
30	240	278	300	225
	169	33	279	>320
		50	210	25
		320	311	
		232	208	
45		>195	>273	255
		162	>230	185
		290	>210	>348

^aUsed as-machined 2219-T87.

> Specimen failed at other than anodize surface.

stripping and reanodizing under production conditions was also examined. The results of Part 3, the production anodizing comparison, are summarized in Tables A12, A13, A14, and A15, and in Figures A5 and A6.

Specimens were anodized between 3/1/72 and 3/23/72. Over the period of the study measured sealed coating weights varied between 2.6 (240) and 3.3 gm/m² (304 mg/ft²), while measured PCH values varied between 21 and 34 percent. The measured salt spray resistances (Table A14) exceeded the 336 hr MIL Spec. requirement for all of the panels with a 10 min. seal. On 3/20/72 and 3/23/72 unsealed panels were produced along with sealed panels for comparison purposes. The salt spray resistance of the unsealed panels was in both cases less than 60 hours.

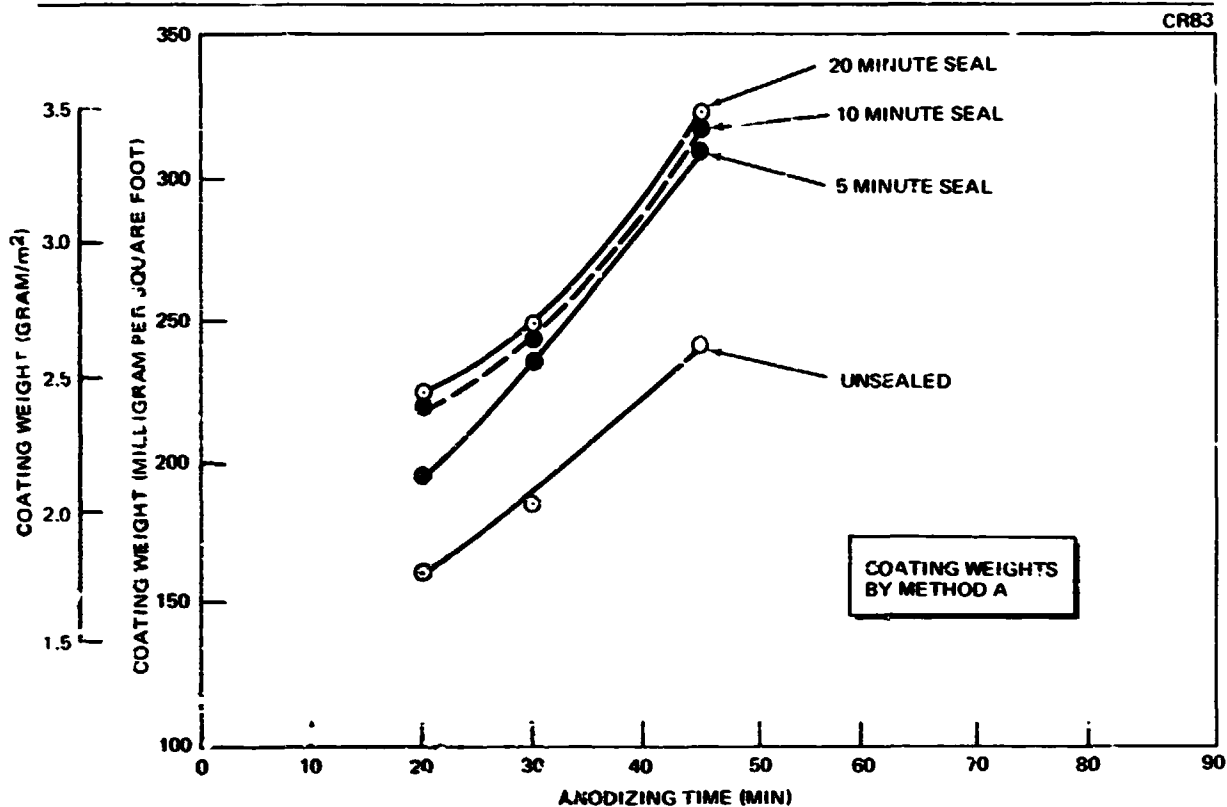


Figure A1. Coating Weight Versus Anodizing and Seal Time, 2219-T87, Machined Surface

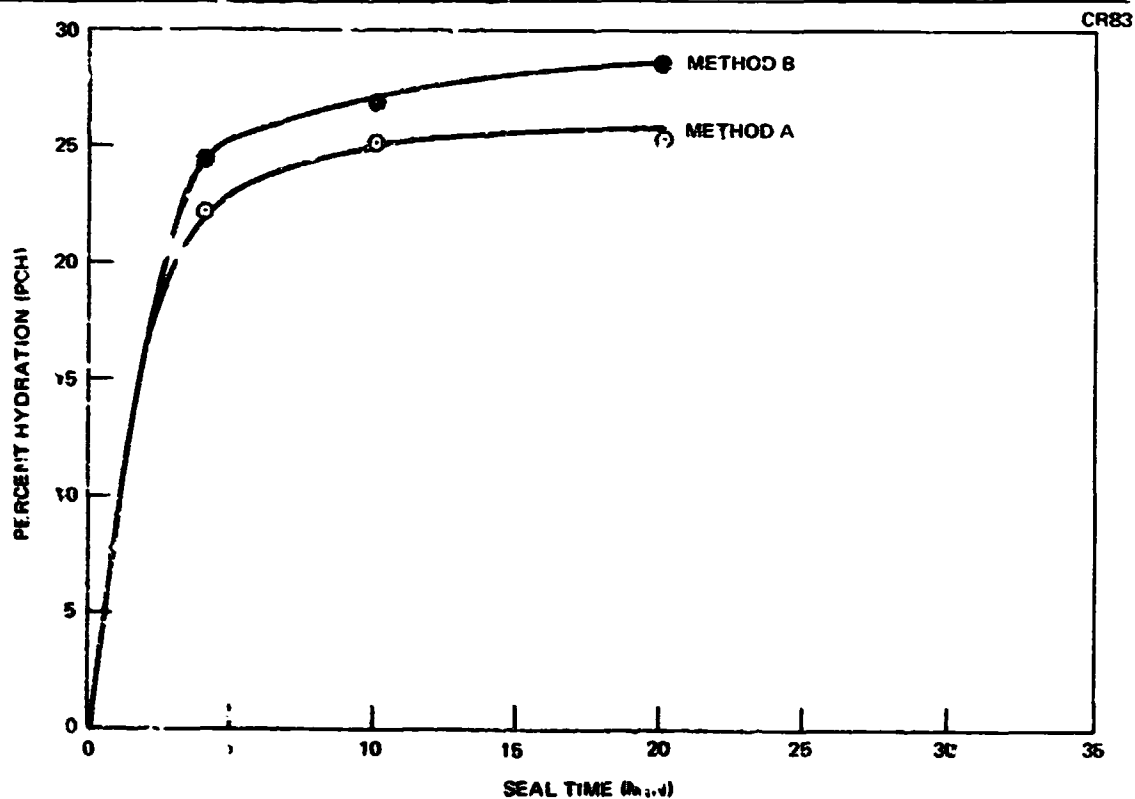


Figure A2. 20-Minute Anodize, 2219-T87, Machined Surface: Percent Hydration Versus Seal Time

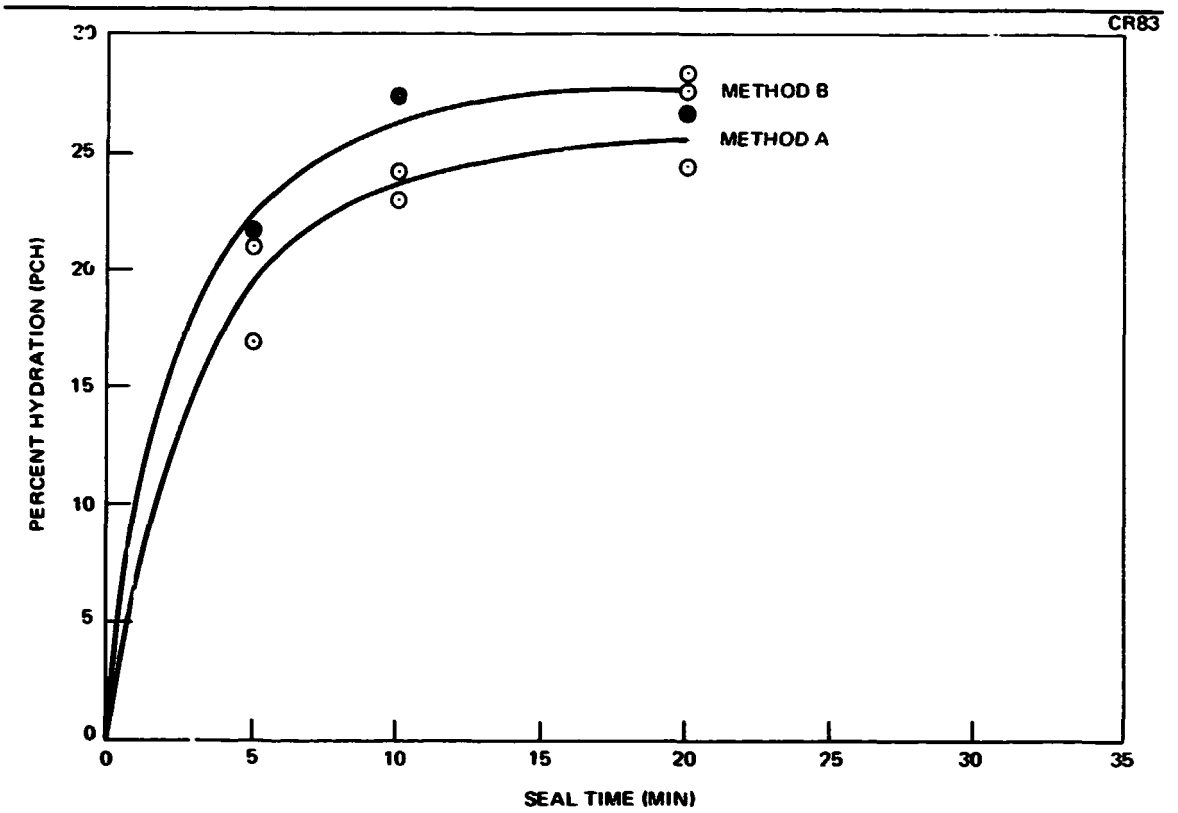


Figure A3. 30-Minute Anodize, 2219-T87, Machined Surface: Percent Hydration Versus Seal Time

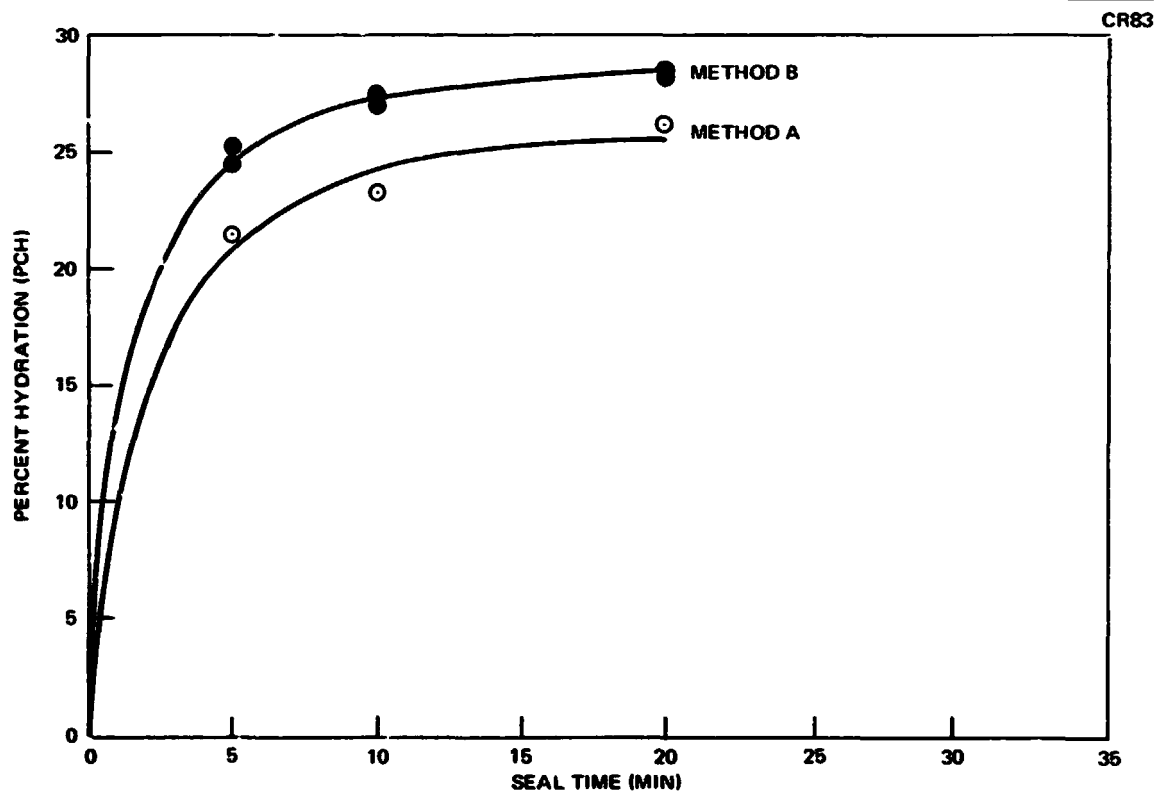


Figure A4. 45-Minute Anodize, 2219-T87, Machined Surface: Percent Hydration Versus Seal Time

Table A12
 PRODUCTION RUN COATING WEIGHT AND PCH
 PROCESS MONITORING FOR 30 MINUTE
 ANODIZING 2219-T87 ALLOY,
 MACHINED SURFACE

Run Number and Process Date	Num- ber of Speci- mens	PCH Method	Unsealed Coat Weight gm/m ² (mg/ft ²)		Seal Time Min	Sealed Coat Weight gm/m ² (mg/ft ²)		PCH
No. 1 3/10/72	2	A	2.09	(194)	10	2.71	(252)	23.0
No. 2 3/03/72	2	A	2.20	(204)	10	2.92	(271)	24.9
	3	B	2.20	(204)*	10	3.09	(287)	29.0
No. 3 3/06/72	2	A	2.16	(201)	10	2.86	(266)	24.3
	2	B	2.16	(201)	10	3.08	(286)	29.5
No. 4 3/08/72	2	A	1.98	(184)	10	2.72	(253)	27.2
	2	B	1.98	(184)*	10	2.96	(275)	33.2
	6	B	1.98	(184)*	10	2.74	(254)**	27.5
No. 5 3/13/72	2	A	2.36	(219)	10	3.14	(292)	25.0
	2	B	2.36	(219)*	10	3.27	(304)	28.0
No. 6 3/16/72	2	A	2.25	(209)	10	2.92	(271)	22.9
No. 7 3/17/72	2	A	2.18	(203)	10	2.85	(265)	23.4
No. 8 3/18/72	2	A	2.23	(207)	10	2.91	(270)	23.1
Special before addition to bath 3/22/72	2	A	2.01	(187)	10	2.59	(241)	22.1
	2	B	2.01	(187)*	10	2.64	(246)**	23.8
No. 9 3/23/72	2	A	2.24	(208)	10	2.94	(273)	23.8
	2	B	2.24	(208)	10	2.85	(265)**	21.5

*Unsealed coating weight from Method A.

**"B" Method specimens sealed at same time as "A" specimens.

Table A13
 -196°C (-320°F) TENSILE STRENGTH:
 PART 3 – PRODUCTION RUN^a

Anodize Date	MN/m ²	(psi)
3/1/72	1.45	(210)
	<u>1.63</u>	<u>(236)</u>
	1.54	(223)
3/3/72	1.16	(168)
	<u>1.35</u>	<u>(196)</u>
	1.26	(182)
3/6/72	0.03	(5)
	<u>1.19</u>	<u>(173)</u>
	0.61	(89)
3/8/72	1.67	(242)
	<u>1.09</u>	<u>(158)</u>
	1.38	(200)
3/12/72	1.08	(156)
	<u>1.45</u>	<u>(210)</u>
	1.26	(183)
3/16/72	1.68	(244,
	<u>1.12</u>	<u>(162)</u>
	1.40	(203)
3/17/72	1.50	(217)
	<u>1.53</u>	<u>(222)</u>
	1.51	(219)

^aA 30 min. anodize with a 10 min. seal was used.

Table A14

**FIVE PERCENT SALT SPRAY RESISTANCE TEST RESULTS: 30 MINUTE ANODIZED 2219-T87
(MACHINED SURFACE), "PRODUCTION RUN" EVALUATION**

Process Date	Num-ber of Specimens	Seal Time, Min	Unsealed*		Sealed*		Percent Hydration (PCH)		Salt Spray Resistance (hr)	
			Coat Weight gm/m ² (mg/ft ²)	Coat Weight gm/m ² (mg/ft ²)	Coat Weight gm/m ² (mg/ft ²)	Coat Weight gm/m ² (mg/ft ²)	Method A	Method B	Mini-mum	Maxi-mum
3/01/72	4	10	2.09	(194)	2.71	(252)	23.0	--	500	500
3/03/72	2	10	2.20	(204)	2.92	(271)	24.8	29.0	336	336
3/06/72	2	10	2.18	(202)	2.86	(266)	24.2	29.4	600	600
3/08/72	2	10	1.98	(184)	2.72	(252)	26.9	33.2	600	950
3/13/72	2	10	2.36	(219)	3.14	(292)	25.0	28.0	672**	
3/16/72	2	10	2.25	(209)	2.92	(271)	22.8	--	672**	
3/17/72	2	10	2.18	(203)	2.86	(265)	23.3	--	672**	
3/20/72	2	10	2.23	(207)	2.90	(270)	23.1	--	672**	
3/23/72	2	10	2.24	(208)	2.94	(273)	23.8	21.5	528**	

*Coating weights listed are Method A values.
 **No record failure to data. Test hours as of 4/21/72.

Table A15

EFFECT OF "REWORKING" PREVIOUSLY ANODIZED 2219-T87

Number of Specimens	Anodize Time	Seal Time	Coat Weight, gm/m ² (mg/ft ²)		Percent Hydration		Salt Spray Resistance (hr)	
			Initial	Rework	Initial	Rework	Initial	Rework
4	30 min	10 min	2.72	(252)	2.75	(255)	23.0	22.3
							500 hr	600 hr

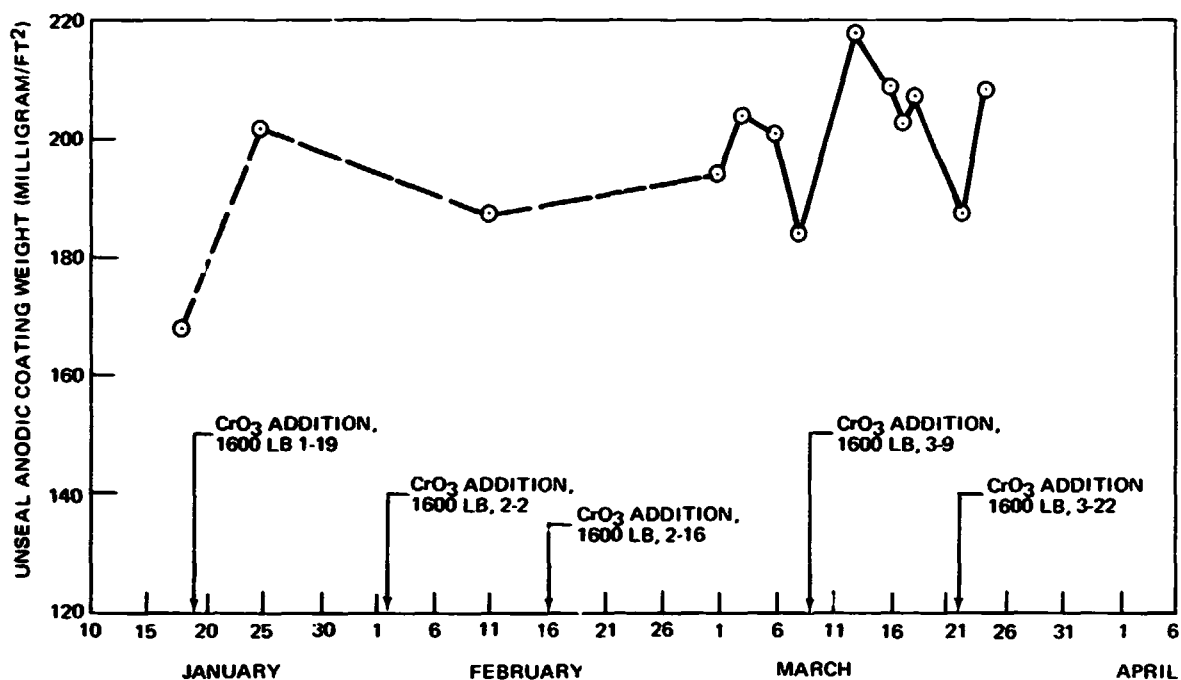


Figure A5. Unsealed Anodic Coating Weights, Production Line, Unit 134 from January to March, 1972

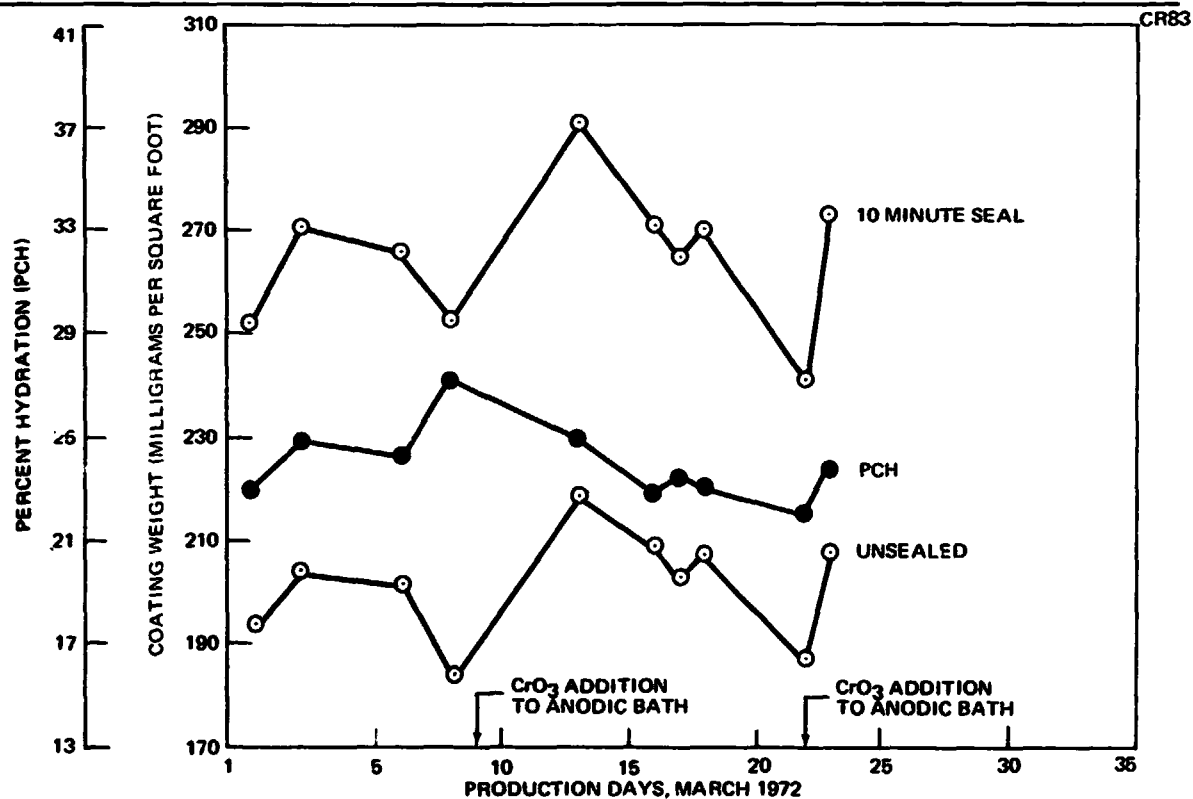


Figure A6. Production Run Test: Coating Weight and Percent Hydration for 2219-T87 Processed During March 1972

The rework panels were anodized, stripped, and then reanodized and tested. One set of panels was initially anodized on 3/1/72 and reanodized on 3/3/72. The other set was initially anodized on 3/3/72 and was reworked on 3/6/72. The consolidated anodize data on these panels are reported in Table A12. The tensile strengths for the panels are reported below.

Rework Panel Tensile Strength
at -196°C (-320°F)
MN/m² (psi)

	2.07	(300)
	1.81	(262)
	1.76	(255)
	<u>1.32</u>	<u>(192)</u>
Average	1.74	(252)

It was considered important based on previous Saturn experience to characterize both the alloy batch properties and the anodized surface characteristics for reference in possible future investigations. For this reason scanning electron microscope (SEM) pictures were taken of an as-machined anodized 2219-T87 surface. They are shown in Figure A7. The photographs are of the same area of the same specimen at three levels of magnification. In Figure A7 (a) shows machining marks and the general appearance of the surface, (b) shows a combination of machining marks and anodic coating and (c) shows primarily the appearance of the anodic coating. The specimen surface is representative of the surfaces of panels used for tensile bond testing during the second half of Phase II. It was anodized for 30 min in a production operation using a 10 min seal (specimen code 30XB10-64).

As a preliminary characterization of the alloy batch used, uniaxial tensile tests were conducted on flat tensile dogbone specimens made from machined panels of the 2219-T87 aluminum batch. The panels were similar to those used for tensile bond testing. The data are presented in Table A16. Table A17 gives a summary of characterization data obtained on surface roughness of the alloy panels used.

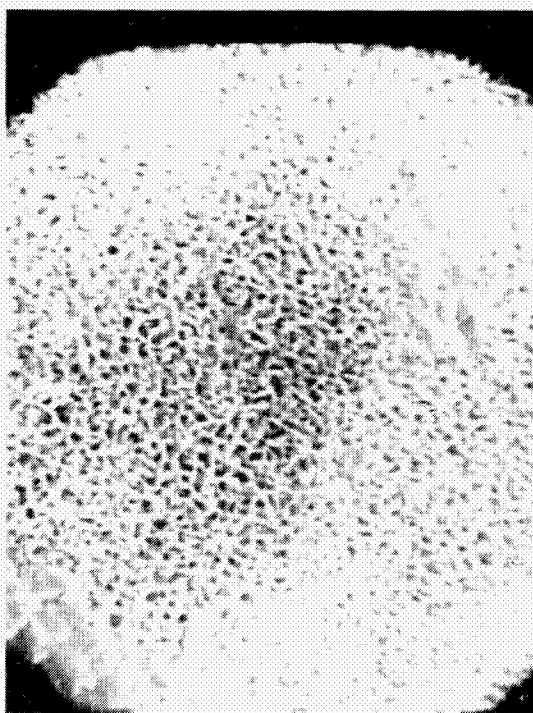
An emission spectrograph was also produced which indicated that the 2219-T87 contained 6.0% Cu, 0.26% Mn, 0.18% Fe, 0.13% Zr, 0.10% V, 0.095% Si, 0.014% Mg, and 0.05% Ti. These concentrations were within the composition limits specified by ALCOA.



580X (a)



2300X (b)



11,600X (c)

Figure A7. As-Machined Anodized 2219-T87

Table A16
TENSILE PROPERTIES, 2219-T87

Specimen	Finish	Yield Strength,		Ultimate Strength,		Elongation, Percent in 2 in.
		MN/m ²	(psi)	MN/m ²	(psi)	
1	Bare, Machined Surface	384.3	(55,725)	475.5	(68,970)	8
2		386.1	(55,985)	473.7	(68,705)	8
3		389.8	(56,525)	477.2	(69,210)	8
4		383.9	(55,595)	476.1	(69,045)	8
5		<u>382.8</u>	<u>(55,515)</u>	<u>475.4</u>	<u>(68,945)</u>	<u>8</u>
Average		385.3	(55,870)	475.6	(68,975)	8
1	30 Minute	386.8	(56,085)	475.6	(68,975)	8
2	Anodized	388.3	(56,310)	477.0	(69,175)	8.5
3	Machined	386.5	(56,040)	474.6	(68,840)	8.5
4	Surface	388.7	(56,370)	475.7	(68,990)	8.5
5		<u>382.8</u>	<u>(55,515)</u>	<u>476.6</u>	<u>(69,115)</u>	<u>8.5</u>
Average		386.6	(56,065)	475.9	(69,020)	8.5

Table A17
SURFACE ROUGHNESS MEASUREMENT OF TYPICAL
2219-T87 MACHINE SURFACE

After Alkaline Cleaned RMS ^a μm (μ in.)				After 30 Min Anodize RMS ^a μm (μ in.)			
High		Low		High		Low	
1.26	(49.5)	0.99	(39.0)	1.11	(43.5)	0.93	(36.5)
1.25	(49.0)	1.04	(41.0)	1.26	(49.5)	1.07	(42.0)
1.25	(49.0)	1.04	(41.0)	1.17	(46.0)	0.95	(37.5)
1.57	(62.0)	1.16	(45.5)	1.32	(52.0)	1.13	(44.5)
1.23	(48.5)	0.96	(38.0)	1.17	(46.0)	0.99	(39.0)
<u>1.26</u>	<u>(49.5)</u>	<u>0.99</u>	<u>(39.0)</u>	<u>1.09</u>	<u>(43.0)</u>	<u>0.91</u>	<u>(36.0)</u>
Average	1.30 (51.2)	1.03	(40.6)	1.19	(46.7)	1.00	(39.2)

^a Average of 4 readings.